



Automatic Polishers

Suggested Procedures for Grinding & Polishing Various Alloys



Foreword

The following automated procedures were developed in the metallographic laboratory at LECO CORPORATION. The procedures are reproducible but by no means absolute. Variations can and are expected to be tried by individuals processing samples by automated means. These parameters also are useful when manual preparation is being done.

The number of specimens prepared simultaneously can vary from as few as three, and up to twelve, depending on the size of the mounts. Unmounted specimens can be processed equally as well as mounted specimens, but because of the larger metal surface areas, the time used for the grinding steps may need to be increased, or by introducing a coarser grit size at the initial grind.

The lubrication oil used with diamond compound or diamond grinding discs and the alumina slurry used for final polishing are introduced from the reservoirs contained in the unit. Alumina slurry containing acids (chromic, oxalic, ammonium hydroxide-hydrogen peroxide, etc.) usually are administered by hand. If the same material is being processed on a continuing basis, acids can be mixed with the slurries contained in the reservoirs.

After polishing wheels have been initially charged with polishing media and wetted with the appropriate lubricants, the frequency of dispersion should be approximately every 60 seconds for the microid extender and every 30 seconds for the alumina slurry.

Although it is preferable to process like alloys together, intermixing of alloys using the same procedures can be successfully done. For example, aluminum alloys can be processed along with copper alloys—both procedures are essentially the same; different grades of steels can be done together, tungsten carbide samples with aluminum oxide samples, etc.

Certain prerequisites are required before using the following procedures. Mounted specimens should be placed directly in the holder without hand grinding—parallelism is already established. Flashing may be removed from mounts by "walking" the edges around a grinding paper. When processing unmounted samples, all burs need to be removed. Coplanarity between samples and holders needs to be established during initial grind before proceeding to succeeding steps. See section under "Helpful Hints", page 55.

Having a problem? Call the Metallographic Laboratory for assistance: 269-982-2385 or 269-982-2266. Visit www.leco.com for more information on our products.

LECO would like to thank Dr. Lee Dillinger for his contributions to this project.



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Aluminum

Using CAMEO® Magnetic Discs

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	60	CW	75	20	150

Polishing

CAMEO Gold Disc/ 6 micron CAMEO suspension/ microid extender	120	CW	75	20	150
CAMEO White-FAS Disc/ 3 micron diamond compound/ microid extender	180	CW	75	45	150
1 micron diamond compound/red felt cloth/microid extender	60	CW	75	45	150
0.05 micron colloidal silica/Imperial Cloth	60	CW	75	35	150



Aluminum and Aluminum Alloys

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

1 micron diamond compound/ red felt cloth/microid extender	300	250	30
colloidal silica/Imperial Cloth (wetted)	60	150	15

Remarks

A few drops of a solution composed of 50 ml ammonium hydroxide and 5 ml hydrogen peroxide dropped on the final polishing wheel will chemically polish and remove fine alumina scratches.

Suggested Etchants

Kellers Reagent
Immerse

Welds or Macrostructure
1 part H₂O, 1 part HCl, 1 part HF
15% Aqueous NaOH (Immerse)

2090 Aluminum with Li-Cu-Zr

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	30
320	60	300	30
600	60	300	30

Polishing

3 micron diamond compound/ silk cloth/microid extender	120	250	40
1 micron diamond compound/ red felt cloth/microid extender	240	250	45
colloidal silica/Imperial Cloth/ water	60	150	25

Suggested Etchants

Kellers Reagent



Aluminum, As-Cast

Using CAMEO[®] Magnetic Discs

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	60	CCW	75	3	150

Polishing

CAMEO Gold Disc/ 6 micron CAMEO suspension/ microid extender	120	CCW	75	3	150
3 micron diamond paste/ Pan W cloth/ microid extender	180	CCW	75	3	150
1 micron diamond compound/red felt cloth/microid extender	60	CCW	75	3	150
0.05 micron colloidal silica/Imperial Cloth	60	CCW	75	3	150

Aluminum, As-Cast

Sectioning

Bandsaw, Al_2O_3 Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	30
320	60	300	30
600	250	45	

-Ultrasonically Clean-

Polishing

3 micron diamond compound/ silk cloth/microid extender	300	250	30
1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/Imperial Cloth/ water	60	150	20

Etchants

Barkers

Electrolytic: 1A, 2 to 3 min., view under polarized light

Kellers

Immerse



Aluminum, As-Cast

Sectioning

Bandsaw, Al_2O_3 Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media
(Rough Procedure)

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	30
600	60	300	30

-Ultrasonically Clean-

Polishing

1 micron diamond compound/ red felt cloth/microid extender	120	250	20
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Macro Etch

15% Aqueous NaOH
Immerse 10 minutes

Barkers

Electrolytic: 1A, 2 to 3 min., view under polarized light

Aluminum, Unmounted Samples

(Unmounted Samples up to 1.5 inches in diameter.

Larger automatic polishers only.)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
120	60	300	32
180	60	300	32
240	60	300	32
320	60	300	32
400	60	300	32
600	60	300	32

Polishing

6 micron diamond compound/ red felt cloth/microid extender	240	250	30
1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/Imperial Cloth/ wetted	180	100	0

Etchants

Kellers



Aluminum Oxide

(Ceramic)

Sectioning

Diamond Cutoff Wheel

Mounting

Castable Mounting Media. Avoid compression mounting unless samples are absolutely flat. Add Pelletized Al_2O_3 (3 to 5 micron) to equate grinding characteristics.

Grinding

Diamond Grinding Disc Size (microns)	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
74/water	120	300	35
40/water	120	300	35
20/water	120	300	35

Polishing

6 micron diamond compound/ silk cloth/microid extender	360	250	35
3 micron diamond compound/ silk cloth/microid extender	360	250	35
1 micron diamond compound/ silk cloth/microid extender	120	250	35
colloidal silica/Imperial Cloth/ water	30	150	20

Etchants

Boiling Phosphoric Acid

Wait until boiling action subsides before placing sample in etchant, 5 to 10 min.

Aluminum Oxide

(Sparkplug, Ceramic)

Sectioning

Diamond Cutoff Wheel

Mounting

Castable, add pelletized Al_2O_3 to castable to equate grinding characteristics.

Grinding

Diamond Grinding Disc Size (microns)	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
125/water	120	300	30
74/water	120	300	30
40/water	120	300	30
20/water	120	300	30

Polishing

9 micron diamond compound/ silk cloth/microid extender	120	250	30
3 micron diamond compound/ silk cloth/microid extender	120	250	30
1 micron diamond compound/ red felt cloth/microid extender	180	250	25
colloidal silica/Imperial Cloth/ water	120	150	10

Etchants

Boiling Phosphoric Acid

Wait until vigorous boiling action subsides before placing sample in etchant, 5 to 10 min.



Aluminum Silicon Carbide

Using CAMEO® Magnetic Discs

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CCW	25	75	CCW	200
CAMEO Platinum #2	2:00	CCW	25	75	CCW	200

Pre-Polishing

CAMEO Silver Disc/
6 micron CAMEO
suspension/microid
extender

2:00	CCW	25	75	CCW	200
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Polishing

3 micron premium
suspension/ultra silk/
microid extender

10:00	CCW	40	100	CCW	200
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1 micron premium
suspension/red felt/
microid extender

1:00	CCW	20	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

1:00	CCW	20	75	CCW	150
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Babbitt on Nickel Aluminide on Steel

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Thermosetting Resins or Castables

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	40
320	30	300	40
600	30	300	40

Polishing

6 micron diamond compound/ nylon/microid extender	180	250	30
1 micron diamond compound/ red felt cloth/microid extender	90	250	30
colloidal silica/wetted Imperial Cloth	60	150	15

Remarks

Polishing can terminate after 1-micron diamond polish, depending on degree of polish desired.

Etchants

2% Nital to show steel-aluminide interface



Barium Titanate

(Electronic Ceramic, Capacitors)

Sectioning

Mount First, Diamond Wafering Blade

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
320 ^(a)	120	300	40
400	60	300	40
600	60	300	40

^(a)Continue grinding until center of capacitor is reached

Polishing

3 micron diamond compound/ silk cloth/microid extender	300	200	30
1 micron diamond compound/ red felt cloth/microid extender	120	200	20
colloidal silica/Imperial Cloth/ wetted cloth	60	150	10

Etchants

Boiling Phosphoric Acid

Wait until vigorous boiling action subsides before placing sample in acid,
5 to 10 minutes.

Beryllium

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/Imperial Cloth/ wetted cloth	120	150	10

Remarks

A few drops of 10% Oxalic Acid dropped on the final polishing wheel will facilitate removal of fine polishing scratches. Microscopic examination under polarized light will reveal grain structure and any mechanical twins that have been introduced during sectioning. If mechanical twins are observed, repeating the polishing sequence will remove them.



Boron Filaments in Magnesium Matrix

Sectioning

SiC Cutoff Wheel/Coolant, or Diamond Wafering Blade

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
15 micron diamond grinding disc/water	120	300	40

Polishing

6 micron diamond compound/ silk cloth/microid extender	300	250	30
1 micron diamond compound/ silk cloth/microid extender	60	250	25
1 micron diamond compound/ red felt cloth/microid extender	60	150	25
colloidal silica/ wetted Imperial Cloth	60	150	15

Remarks

Depending on the degree of polish desired, polishing could terminate after any of the diamond polishings. For example, the filaments are very flat and smooth after the 6-micron polish; however, the matrix has fine scratches.

Etchants

Magnesium can be examined with polarized light.

5% Nital

Immerse

Brass

Using CAMEO® Magnetic Discs

Sectioning

SiC or Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	60	CCW	75	25	150

Polishing

CAMEO Gold Disc/ 6 micron CAMEO suspension/ microid extender	120	CCW	75	25	150
CAMEO White-FAS Disc/ 3 micron diamond compound/ microid extender	180	CCW	75	45	150
1 micron diamond compound/red felt cloth/microid extender	60	CCW	75	45	150
0.05 micron colloidal silica/Imperial Cloth	60	CCW	75	35	150



Cadmium on Nickel on Steel

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/ red felt cloth/microid extender	180	250	30
0.05 micron Al ₂ O ₃ /Lecloth/ water	60	150	30

Remarks

Water may darken cadmium coatings. Replace water with ethyl alcohol during final polishing step with alumina.

Etchants

(Steel) 2% Nital
Immerse

Carbon Reinforcements in Epoxy Resin

(Composites)

Sectioning

Al₂O₃ or SiC Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
180	60	300	35
320	60	300	35
600	60	300	35

Polishing

6 micron diamond compound/ silk cloth/microid extender	300	250	30
1 micron diamond compound/ red felt cloth/microid extender	120	250	30
colloidal silica/Imperial Cloth/ water	60	150	25



Cast Iron

(Fixed Sample)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
180 grit	60	CCW	100	40	200
320 grit	60	CCW	100	40	200
600 grit	60	CCW	100	40	200

Polishing

3 micron diamond compound/silk cloth/ microid extender	180	CCW	100	40	200
1 micron diamond compound/red felt cloth/ microid extender	60	CCW	100	40	200
0.05 micron colloidal silica/Imperial Cloth	60	CCW	75	30	150

Cast Iron

Using CAMEO[®] Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	60	CW	75	25	150

Polishing

CAMEO Silver Disc/
6 micron CAMEO suspension/
microid extender

120

CW

75

25

150

CAMEO White-FAS Disc/
3 micron diamond paste/
microid extender

180

CW

75

45

150

1 micron diamond
compound/red felt
cloth/microid extender

60

CW

75

45

150



Cast Iron

(Gray)

Sectioning

Bandsaw or Al_2O_3 Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	40
320	60	300	40
600	60	300	40

Polishing

1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/ wetted Imperial Cloth	120	150	15

Remarks

Ferritic gray cast iron may require an etch-polish.
Pearlitic gray cast iron can skip the final polishing step.

Etchants

2% Nital
 Ferritic grades
4% Picral
 Pearlitic or heat treated grades

Cast Iron

(Ductile & Malleable)

Sectioning

Bandsaw or Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	40
320	60	300	40
600	60	300	40

Polishing

3 micron diamond compound/ silk cloth/microid extender	180	250	30
1 micron diamond compound/ red felt cloth/microid extender	60	250	30
colloidal silica/wetted Imperial Cloth	60	150	15

Remarks

This procedure can be used with gray cast irons also. The shorter time with the final polishing step eliminates relief of the graphite nodules. Pearlitic ductile and malleable cast irons may not require the final polish. Observe under polarized light for clarity of cross nichols in the graphite nodules.

Etchants

4% Picral or 2% Nital.



Cast Iron with Enamel Coating

Sectioning

Al₂O₃ or SiC Cutoff Wheel/Coolant

Mounting

Diallyl Phthalate

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
220 mesh diamond disc/water	180	150	40
320 SiC/water	60	300	40
400 SiC/water	60	300	40
600 SiC/water	60	300	40

Polishing

3 micron diamond compound/ silk cloth/microid extender	300	250	30
Finish-Pol ^(a) /wetted Lecloth	60	100	20

^(a)Finish-Pol polishing slurry containing cerium oxide, gamma alumina and other rare earth oxide

Remarks

Encapsulating specimen in aluminum foil before mounting will give excellent contrast between the enamel coating—mounting media interface.

Etchants

2% Nital
Cast Iron

Ceramic

Sectioning

Diamond Low-Deformation Saw

Mounting

Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
20 micron diamond spot pattern	600	CCW	75	25	150

Polishing

9 micron diamond compound/silk cloth/ microid extender	600	CCW	75	25	150
3 micron diamond compound/silk cloth/ microid extender	600	CCW	75	25	150
0.05 micron colloidal silica/Imperial Cloth	120	CCW	75	25	150



Clinker Samples

Using CAMEO® Magnetic Discs

Sectioning

Diamond Blade

Mounting

Epoxy

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CW	35	75	CCW	200
CAMEO Platinum #2	2:00	CW	35	75	CCW	200

Polishing

3 micron premium
suspension/ultra silk/
microid extender

3:00	CW	35	100	CCW	200
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0.05 micron gamma B
alumina powder/
microid extender/
Lecloth

1:00	CW	30	75	CCW	150
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Remarks

Rinse only with ethyl alcohol between preparation steps to alleviate staining.

Etchants

2% Nital

Immerse distilled water 104-122°F

Immerse

Coal

Sectioning

Diamond Low-Deformation Saw

Mounting

Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
320 grit	15	CCW	100	35	200
600 grit	30	CCW	100	35	200

Polishing

3 micron diamond compound/silk cloth/ microid extender	120 to 180	CCW	100	35	200
0.05 micron colloidal silica/Imperial Cloth	45	CCW	75	35	150



Coal and Coke

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
400	60	300	40
600	60	300	40

Polishing

0.05 micron Al₂O₃/Lecloth/
water/10% Cr₂O₃^(a)

240

300

40

^(a)Chromic Acid—100 ml H₂O, 10g Cr₂O₃

Remarks

Chromic acid is manually introduced to the polishing wheel by means of squeeze bottle or eyedropper.

Examine coke under polarized or sensitive tint illumination.

Cobalt Alloy

Using CAMEO® Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite or Epoxy

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CCW	35	75	CCW	200

Pre-Polishing

CAMEO Silver Disc/
6 micron CAMEO
suspension/microid
extender

3:00	CCW	35	75	CCW	200
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Polishing

3 micron premium
suspension/ultra silk/
microid extender

3:00	CCW	40	100	CCW	200
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1 micron premium
suspension/red felt/
microid extender

1:00	CCW	40	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

1:00	CCW	30	75	CCW	150
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Concrete

Sectioning

Diamond

Mounting

Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
74 micron diamond spot pattern	60	CW	100	30	150
20 micron diamond spot pattern	60	CCW	100	30	150
10 micron diamond spot pattern	60	CCW	100	30	150

Polishing

6 micron diamond compound/nylon/ microid extender	180	CCW	100	30	150
1 micron diamond compound/red felt cloth/ microid extender	60	CCW	100	20	150

Copper

Using CAMEO® Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	60	CCW	75	35	150

Polishing

CAMEO Gold Disc/ 6 micron CAMEO suspension	120	CCW	75	35	150
3 micron diamond compound/ Pan W/microid extender	180	CCW	75	40	200
1 micron diamond compound/red felt cloth/microid extender	60	CCW	75	40	200
0.05 micron colloidal silica/Imperial Cloth/ water	90	CCW	75	35	150



Copper

(Pure, OFHC)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
240	60	300	40
600	60	300	40

Polishing

1 micron diamond compound/ red felt cloth/microid extender	180	250	30
Ferric oxide slurry/Lecloth	60	150	20

Remarks

The Ferric Oxide final polish is recommended for microscopic examination in the as-polished condition; however, it leaves a passive film which is inert to etching. A few turns on an alumina polishing cloth will remove the passivity for etching purposes. Gamma alumina (0.05μ) can be used as the final polishing medium. The addition of a few drops of a solution composed of 50 ml NH₄OH and 5 ml H₂O₂ will facilitate polishing.

Etchants

50 ml ammonium hydroxide (NH₄OH), 5 ml hydrogen peroxide (30%) H₂O₂

Immerse

NOTE: If etchant is too fast, add 50 ml H₂O.

To differentiate between cuprous oxide and copper sulfide inclusions, examine in the as-polished condition under polarized light. Cuprous oxide will be red, copper sulfide will remain dark. Both are medium gray with brightfield illumination.

Copper Alloys

(Brasses)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	40
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

3 micron diamond compound/
silk cloth/microid extender

180

250

30

1 micron diamond
compound/red felt cloth/
microid extender

180

250

35

0.05 micron gamma
alumina/Lecloth/water^(a)

180

150

40

^(a)Optional—To keep lead clean and metallic looking.
Polishing may terminate with 1 micron diamond polish.

Etchants

50 ml H₂O, 50 ml Na₄OH, 5 ml H₂O₂
Immerse



Copper Alloys

(Bronzes)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	35
320	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/ red felt cloth/microid extender	180	250	30
colloidal silica/ wetted Imperial Cloth	120	150	15

Etchants

1 g NaCl, 95 ml H₂O, 2 ml 20% chromic acid, 2 ml H₂SO₄
Immerse

95 ml ethyl alcohol, 5 g ferric chloride, 10 ml HCl
Immerse

Copper–Beryllium Alloys

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	40
320	60	300	40
600	60	300	40

Polishing

1 micron diamond compound/ red felt cloth/microid extender	240	250	30
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Ferric Oxide + 10% Cr ₂ O ₃ ^(a) / Lecloth	120	150	30
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^(a) 10% Cr₂O₃ = 100 ml H₂O, 10 g Cr₂O₃

Remarks

Ferric Oxide solution is added to the polishing wheel manually.

Etchants

95 ml ethyl alcohol, 5 g ferric chloride, 10 ml HCl
Immerse



Copper Alloys with Niobium Filaments

Sectioning

Diamond Wafering Wheel

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
120	60	300	40
320	60	300	40
400	60	300	30
600	60	300	30

Polishing

9 micron diamond compound/ silk over silk/microid extender	300	250	30
1 micron diamond compound/ silk over silk/microid extender	300	200	30
0.05 micron Al_2O_3 /Lecloth/ water	300 to 600	100	30

Remarks

If niobium fibers are in relief after 300 seconds polishing with alumina, longer time will flatten the fibers without over-polishing the copper matrix.

Etchants

50 ml NH_4OH , 5 ml H_2O_2
Immerse

If etching too fast, add 50 ml H_2O

Glass

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	40
240	120	300	40
320	120	300	40
400	60	300	40
600	60	300	40

Polishing

9 micron diamond compound/ red felt cloth/microid extender	300	250	30
1 micron diamond compound/ red felt cloth/microid extender	120	250	30
Finish-Pol ^(a) /wetted Lecloth	120	150	15

^(a) *Rare earth oxides in suspension*



Glass

Sectioning

Diamond Low-Deformation Saw

Mounting

Unmounted or Castable Mounting Media

Grinding

	Time (sec.)	Speed (RPM)	Pressure (psi)
180 grit SiC	30	300	50
320 grit SiC	30	300	50
600 grit SiC	30	300	50
800 grit SiC	60	300	50
1200 grit SiC	60	300	50

Polishing

6 micron diamond lapping film	600	300	50
0.05 micron colloidal silica/Imperial Cloth	120	150	50

Glass/Unmounted Samples

Using CAMEO® Magnetic Discs

Sectioning

Diamond Blade

Mounting

N/A

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	4:00	CW	35	100	CCW	200
CAMEO Platinum #2	2:00	CW	35	100	CCW	200
CAMEO Platinum #3	2:00	CW	35	100	CCW	200
CAMEO Platinum #4	2:00	CW	35	100	CCW	200

Polishing

3 micron premium
diamond suspension/
ultra silk/
microid extender

6:00	CW	35	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

2:00	CW	25	75	CCW	150
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Gold

(Au on Cu on Steel or Ni Substrate)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

3 micron diamond compound/ silk cloth/microid extender	180	250	40
1 micron diamond compound/ silk cloth/microid extender	180	250	40
1 micron diamond compound/ red felt cloth/microid extender	60	250	45
colloidal silica/ wetted Imperial Cloth	60	100	15

Etchants

Gold: 1 part 10% ammonium persulfate, 1 part 10% potassium cyanide
Immerse or swab

Incoloy/Inconel

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	35
320	60	300	35
600	60	300	35

Polishing

3 micron diamond compound/ silk cloth/microid extender	180	250	30
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1 micron diamond compound/ red felt cloth/microid extender	60	250	30
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0.05 micron gamma alumina/ wetted Lecloth or colloidal silica/wetted Imperial Cloth	60	250	30
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Remarks

Addition of 10% chromic acid to alumina polishing wheel will facilitate polishing and help remove disturbed metal, particularly when the material is in the annealed condition.

Etchants

10% oxalic acid, electrolytic, 1A, 5 sec.

Glyceregia: 30 ml glycerine, 30 ml HCl, 10 ml HNO₃
Swab



Inconel

Using CAMEO® Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite or Epoxy

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CW	35	75	CCW	200
CAMEO Platinum #2	2:00	CW	35	75	CCW	200

Polishing

6 micron CAMEO
suspension/ultra silk/
microid extender

3:00	CW	40	100	CCW	200
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1 micron premium
suspension/red felt/
microid extender

0:30	CW	35	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

0:30	CW	30	75	CCW	150
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Iron Base Precipitation Hardening Alloys

(A286)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	40
320	60	300	40
600	30	300	40

Polishing

1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/wetted Imperial Cloth	60	150	25

Remarks

Polishing can terminate with the diamond polish.

Etchants

2% Nital

4% Picral



Lead

Sectioning

Microtome Best. Al_2O_3 Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
used 320	60	200	10-15
used 400	60	200	10-15
used 600	60	200	10-15

Polishing

1 micron diamond compound/ red felt cloth/microid extender	300	150	10
colloidal silica/ wetted Imperial Cloth	180	100	—

Remarks

Lead has a tendency for recrystallization during preparation. Etch-polishing several times will eliminate the recrystallization.

Etchants

60 ml acetic acid, 20 ml H_2O_2 , 1 ml HCl
Immerse

NOTE: Responds well to chemical polishing, fine scratches removed.

Magnesium Casting

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Cold Mount, Acrylic, or Bakelite

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
180 Grit SiC	1:00	CW	40	100	CCW	200
320 Grit SiC	1:00	CW	40	100	CCW	200
600 Grit SiC	1:00	CW	40	100	CCW	200

Polishing

3 micron premium
suspension/ultra silk/
microid extender

3:00	CW	40	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

2:00	CW	30	75	CCW	150
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Molybdenum

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/
red felt cloth/microid extender

300

250

30

colloidal silica/
wetted Imperial Cloth

120

150

15

Remarks

View with polarized light.

Etchants

Murakamis

Nickel Alloys

(Udimet 700, Hastelloy, Ni-Co, Ni Zn Ferrite)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
600	60	300	35

Polishing

6 micron diamond compound/ silk cloth/microid extender	300	250	32
6 micron diamond compound/ red felt cloth/microid extender	180	250	32
3 micron diamond compound/ silk cloth/microid extender	300	250	32
1 micron diamond compound/ red felt cloth/microid extender	120	250	32
colloidal silica/Imperial Cloth/ water	30	150	15

Etchants

Udimet 700
marbles reagent

Hastelloy
10% Aqueous HCl, Electrolytic, 1A, 5 sec.

Ni-Co Alloy
96 ml HCl, 4 ml HNO₃, Electrolytic, 0.8 A, 2 sec.



Nickel-Base Superalloys

Using CAMEO® Magnetic Discs (Turbine Blades, etc.)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #1 (until flat)	60	CCW	75	35	150
CAMEO Platinum #2	60	CCW	75	35	150

Polishing

CAMEO Silver Disc/ 6 micron CAMEO suspension	120	CCW	75	35	150
CAMEO White-FAS Disc/ 3 micron diamond compound/ microid extender	180	CCW	75	55	200
1 micron diamond compound/red felt cloth/microid extender	60	CCW	75	50	200
0.05 micron colloidal silica/Imperial Cloth/ water	60	CCW	75	35	150

Nickel-Base Superalloys

(Turbine Blades, etc.)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
240	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

3 micron diamond compound/ silk cloth/microid extender	300	250	32
1 micron diamond compound/ red felt cloth/microid extender	120	250	30
colloidal silica/Imperial Cloth/ water	60	150	20

Etchants

Equal parts 10% Sodium Cyanide, 10% Ammonium Persulfate

10% Aqueous HCl, Electrolytic, 1A, 5 sec.



Niobium

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
240	60	300	40
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

9 micron diamond compound/
silk cloth/microid extender

180

250

30

1 micron diamond compound/
nylon/microid extender

180

250

30

0.05 micron Al₂O₃/Lecloth/
water

300

150

30

Remarks

Addition of 10% oxalic to the final polishing step facilitates polishing and removing disturbed metal.

Etchants

50 ml lactic acid, 30 ml HNO₃, 5 ml HF
Swab

Plasma Spray

(WC, CrC, Cr₂O₃, Al₂O₃, Zr₂O₃, Al-Si, etc. Coatings on Ni, Steel, Inconel Substrates)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Epoxide (Compression), Fluorescent Castable

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	35
240	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

3 micron diamond compound/ silk cloth/microid extender	240	250	30
1 micron diamond compound/ red felt cloth/microid extender	60	250	30
colloidal silica/ wetted Imperial Cloth	60	250	15

Remarks

When sectioning, position material so the cutoff wheel enters the plasma coating and exits the substrate.



Plastic

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
320	30	200	5
600	20	200	5

Polishing

15 micron diamond grinding disc	300	100	40
6 micron diamond compound/ Lecloth/microid extender	120	100	25
1 micron diamond compound/ red felt cloth/microid extender	300	200	25

Remarks

Examine with polarized light, darkfield illumination or Nomarski to view surface anomalies.

Plastic

Sectioning

Al₂O₃ Cutoff Wheel

Mounting

Castable Mounting Media (low heat)

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
320 grit	30	CCW	100	30	200
600 grit	30	CCW	100	30	200
1200 grit	30	CCW	100	30	200

Polishing

3 micron diamond compound/nylon/ microid extender	180	CCW	100	30	200
1 micron diamond compound/red felt cloth/ microid extender	30	CCW	100	30	200
0.05 micron colloidal silica/Imperial Cloth	30	CCW	75	25	150



Printed Circuit Board Coupons

(Plated Through Holes)

Note

PCB System required. Follow directions with PCB System for drilling, positioning holes, pinning coupons, loading into silicone mold, and attaching holder.

Mounting

LECOSET 7007 Castable; Pressure vessel for transparent mounts

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
180 grit SiC	1:00	CW	35	100	CCW	200
240 grit SiC	1:00	CW	35	100	CCW	200
320 grit SiC	1:00	CW	35	100	CCW	200
600 grit SiC	1:00	CW	35	100	CCW	200

Polishing

3 micron premium
suspension/ultra silk/
microid extender

3:00	CW	30	100	CCW	200
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1 micron premium
suspension/red felt/
microid extender

0:30	CW	30	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

0:30	CW	30	75	CCW	150
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Remarks

Color the faces of the carbide stops with a permanent marker. Repeat first grinding step 3 times for adequate material removal. Proceed with next steps until permanent marker has been removed from the carbide stops (320 grit step is the target). Finish with 600 grit.

Rene

Sectioning

SiC Abrasive Cutoff Wheel

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (psi)	Wheel Speed (RPM)
60 grit AlO	120	CCW	75	25	150
120 grit SiC	60	CCW	75	20	150
180 grit SiC	60	CCW	75	20	150
240 grit SiC	60	CCW	75	20	150
320 grit SiC	60	CCW	75	20	150
600 grit SiC	60	CCW	75	20	150

Polishing

9 micron diamond compound/
red felt cloth/
microid extender

240	CCW	75	20	150
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0.05 micron colloidal
silica/Imperial Cloth

120	CCW	75	20	150
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Resulfurized Steel

(11XX, 12XX Alloys)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/
red felt cloth/microid extender

240

250

35

colloidal silica/
wetted Imperial Cloth

120

150

15

Remarks

Rinse specimens in alcohol after colloidal silica polish to eliminate staining of inclusions.

Etchants

2% Nital

SiMO, W-SiGe-Mo, SiGe Coatings on Cu or Ni Substrates

Sectioning

Al₂O₃ or SiC Cutoff Wheel/Coolant

Mounting

Epoxide or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
120	60	300	40
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

1 micron diamond compound/ silk cloth/microid extender	180	250	30
Ferric Oxide + 10% Cr ₂ O ₃ /Lecloth	60	100	35

Remarks

Ferric Oxide Slurry: 500 ml H₂O, 20 grams ferric oxide, 15 ml 10% Cr₂O₃, added manually to the polishing wheel.



Silicon

Sectioning

SiC Cutoff Wheel/Coolant—Slowly

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
600	20	75	5

Polishing

3 micron diamond compound/
silk cloth/microid extender

300

100

30

colloidal silica/Lecloth

600

60

5

Etchants

40 ml distilled H₂O, 10 ml HCl, 10 ml H₂O₂
Immerse

100 ml H₂O, 50 g sodium hydroxide

Silicon Carbide

Sectioning

Diamond Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
63–74 micron diamond grinding disc/water	180	200	30
30–40 micron diamond grinding disc/water	180	200	30
10–20 micron diamond grinding disc/water	60	200	30

Polishing

6 micron diamond compound/ silk cloth/microid extender	240	200	30
1 micron diamond compound/ red felt cloth/microid extender	60	200	30
Optional: colloidal silica/ wetted Imperial Cloth	60	150	10

Etchants

Boiling Halls Reagent
15 min.



Silicon Carbide Filaments in Aluminum

Sectioning

Large—SiC Cutoff Wheel/Coolant

Small—Diamond Wafering Blade

Mounting

Diallyl Phthalate

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
15–20 micron diamond grinding disc/water	300	300	30

Polishing

6 micron diamond compound/ silk cloth/microid extender	300	250	30
1 micron diamond compound/ red felt cloth/microid extender	60	250	20
colloidal silica/ wetted Imperial Cloth	60	150	15

Silicon Carbide on Graphite

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
63–74 micron diamond grinding disc/water	120	300	35
10–20 micron diamond grinding disc/water	180	300	35

Polishing

6 micron diamond compound/ silk cloth/microid extender	240	250	40
1 micron diamond compound/ red felt cloth/microid extender	300	200	30

Remarks

The 1 micron diamond polish is quite adequate. Polishing with colloidal silica or gamma alumina will create relief between the hard coating and softer substrate.



Silicon Nitride

Using CAMEO® Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Epoxy or Diallyl Phthalate

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CW	35	75	CCW	200

Pre-Polishing

CAMEO Silver Disc/
6 micron CAMEO
suspension/microid
extender

5:00	CW	35	75	CCW	200
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Polishing

3 micron premium
suspension/ultra silk/
microid extender

10:00	CW	40	100	CCW	200
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Silver

Using CAMEO® Magnetic Discs

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite

Grinding

	Time (min:sec)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (RPM)
CAMEO Platinum #1	2:00	CW	20	75	CCW	200

Pre-Polishing

CAMEO Gold Disc/
6 micron CAMEO
suspension/microid
extender

2:00	CW	20	75	CCW	200
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Polishing

3 micron premium
suspension/PEFA/
microid extender

3:00	CCW	10	100	CCW	200
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1 micron premium
suspension/red felt/
microid extender

1:00	CCW	10	100	CCW	200
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0.05 micron colloidal
silica/imperial cloth

4:00	CCW	10	75	CCW	150
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Etchant

20 ml NH₄OH 20 ml H₂O₂, 10 ml H₂O; swab for 3 to 10 seconds.



Silver with Cadmium Oxide

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

Sic Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
240	40	300	25
600	60	300	25

Polishing

3 micron diamond compound/
silk cloth/microid extender

60

250

25

1 micron diamond compound/
red felt cloth/microid extender

120

250

30

0.05 micron gamma alumina/
Imperial Cloth/water

30

150

20

Etchants

50 ml H₂O, 25 ml NH₄OH, 3 ml H₂O₂

Immerse

Stainless Steel

(Austenitic)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	35
320	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/
red felt cloth/microid extender

240

250

30

colloidal silica/
wetted Imperial Cloth

120

150

15

Etchants

30 ml Glycerine, 30 ml HCl, 10 ml HNO₃
Swab

10% Oxalic, Electrolytic, 1A, 5 to 10 sec.

Retard twin lines (for image analysis of grain size determination); 60% aqueous nitric acid, electrolytic, 0.6 V, platinum cathode, 2 min.



Stainless Steel

(Powder Metal)

Sectioning

Al₂O₃ Cutoff Wheel

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Speed (RPM)	Pressure (psi)
180 Grit SiC	60	300	50
320 Grit SiC	60	300	50
600 Grit SiC	60	300	50

Polishing

3 micron diamond compound/silk cloth/ microid extender	300	200	50
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*0.05 micron colloidal silica/Imperial Cloth/ water	120	150	40
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Steel

Using CAMEO® Magnetic Discs (HRC>30)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #1 (until flat)	60	CW	75	25	150

Polishing

CAMEO Silver Disc/
6 micron CAMEO suspension/
microid extender

120

CW

75

25

150

CAMEO White-FAS Disc/
3 micron diamond
compound

180

CW

75

45

150

1 micron diamond
compound/red felt
cloth/microid extender

60

CW

75

45

150



Steel

Using CAMEO® Magnetic Discs (HRB < 100)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #1 (until flat)	60	CW	75	25	150
CAMEO Platinum #2	60	CW	75	25	150

Polishing

CAMEO White-FAS Disc/ 3 micron diamond compound	180	CW	75	45	150
1 micron diamond compound/red felt cloth/microid extender	60	CW	75	45	150

Steel

(Low to Medium Carbon)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	30
320	60	300	30
600	60	300	30

Polishing

1 micron diamond/ red felt cloth/microid extender	300	250	25
colloidal silica/ wetted Imperial Cloth	120	150	15

Remarks

With a predominately ferritic matrix, etch lightly with 2% nital while samples are still in the holder. Repeat final polishing step.

Etchants

2% Nital for general microstructure and ferrite grain size determinations.

4% Picral for carbide phase only, without etching ferrite grain boundaries.

To fully bring out all ferrite grain boundaries for image analysis, etch 3 sec., in 2% Nital, followed by 3 sec. in 8 g Oxalic Acid, 100 ml H₂O, 5 ml H₂SO₄ and 5 ml H₂O₂.



Steel

(Medium, High Carbon Steels, Low Alloy Steels, Normalized, Annealed, Hardened and Carburized Steels)

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	32
320	60	300	32
600	60	300	32

Polishing

3 micron diamond compound/ silk cloth/microid extender	240	250	30
1 micron diamond compound/ red felt cloth/microid extender	120	250	30
colloidal silica/Imperial Cloth/ water	60	150	20

Etchants

2% Nital

4% Picral for heat-treated alloys

Tantalum

(TA Alloys)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

Polishing

1 micron diamond compound/ red felt cloth/microid extender	300	250	30
colloidal silica/ wetted Imperial Cloth	120	150	15

Etchants

30 ml Lactic Acid, 30 ml HNO₃, 5 ml HF
Swab



Tin

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
used 180	60	200	30
used 320	30	200	30
used 600	30	200	30

Polishing

0.05 micron Al₂O₃/Lecloth/
water + NH₄OH & H₂O₂

300

150

20

Remarks

Do not use fresh grinding discs. Before grinding tin specimens, remove asperites on the discs by manually moving a steel sample over rotating grinding area by having unit in the MANUAL mode.

Sn is very soft and prone to recrystallization during preparation procedures. Etch-polish in one of the following etchants.

Etchants

5% Nital

5% HCl in ethyl alcohol

10% H₂SO₄ in 100 ml H₂O, electrolytic, 1A, 5 sec.

Pull sample from solution with anode still in contact with sample.

Titanium

Using CAMEO[®] Magnetic Discs

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #2 (until flat)	120	CCW	75	35	150

Polishing

9 micron diamond compound/ Silk/microid extender	180	CCW	75	35	150
0.05 micron colloidal silica/Imperial Cloth/ water	90	CCW	75	35	150



Titanium 6-4

Using CAMEO® Magnetic Discs

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Diallyl Phthalate

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #1 (until flat)	120	CCW	75	35	150
CAMEO Platinum #2	180	CCW	75	35	150

Polishing

9 micron diamond compound/
CAMEO White-FAS

Disc	240	CCW	75	45	175
0.05 micron colloidal silica/Imperial Cloth	120	CCW	75	35	150

Titanium

(Pure, Ti-6Al-4V, Ti-8Al-1Mo-1V, and other Ti Alloys)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	40
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

9 micron diamond compound/
silk cloth/microid extender

300

250

30

colloidal silica/
wetted Imperial Cloth

300

150

15

Remarks

10% oxalic acid added to the final polishing step will facilitate polishing.

Etchants

Kroll's reagent

Immerse or swab

30 ml Lactic Acid, 10 ml HNO₃, 2 ml HF



Titanium Alloy with SiC Inserts

Sectioning

Diamond Cutoff Wheel/Coolant

Mounting

Diallyl Phthalate

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
45–64 micron diamond grinding disc/water	until samples are flat	300	40
20–30 micron diamond grinding disc/water	300	300	40
10–15 micron diamond grinding disc/water	300	300	40

Polishing

9 micron diamond compound/ red felt cloth/microid extender	360	250	30
3 micron diamond compound/ silk cloth/microid diamond extender	840	250	30
colloidal silica/Imperial Cloth/ water	120	100	20

Etchants

Krolls Reagent

Tungsten Carbide

Using CAMEO® Magnetic Discs

Sectioning

Diamond Cutoff Wheel/Coolant

Mounting

Epoxide or Diallyl Phthalate (glass-filled)

Grinding

	Time (sec.)	Direction	Head Speed (RPM)	Pressure (lb.)	Wheel Speed (RPM)
CAMEO Platinum #1 (until flat)	60	CW	75	25	150

Polishing

CAMEO Silver Disc/ 6 micron CAMEO suspension/ microid extender	120	CW	75	25	150
CAMEO White-FAS Disc/ 3 micron diamond compound/ microid extender	180	CW	75	50	150
0.05 micron colloidal silica/ Imperial Cloth	60	CW	75	35	150



Tungsten Carbide

Sectioning

Diamond Cutoff Wheel/Coolant

Mounting

Diallyl Phthalate

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
45–64 micron diamond grinding disc/water	180	300	40
20–30 micron diamond grinding disc/water	180	300	40

Polishing

3 micron diamond compound/ silk cloth/microid extender	240	250	35
1 micron diamond compound/ red felt cloth/microid extender	120	250	35
colloidal silica/wetted Imperial Cloth	60	150	15

Remarks

Polishing can be terminated after the 1 micron diamond polish. The gamma alumina polish only serves to give better contrast between the tungsten carbide grains and the cobalt binder.

Etchants

Murakamis Reagent

Tungsten Carbide with Diamonds

(with a cobalt or copper alloy binder)

Sectioning

Diamond Cutoff Wheel/Coolant

Mounting

Diallyl Phthalate

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
100 mesh diamond grinding disc/water	180	200	40
220 mesh diamond grinding disc/water	180	200	40
30 micron diamond grinding disc/water	120	200	40
30 micron diamond compound/ canvas/microid extender	90	200	40

Polishing

0.05 micron Al_2O_3 /Lecloth/water	180	100	30
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Remarks

No more than three specimens should be prepared at one time, as the diamonds contained in the specimens are too severe on the diamond grinding discs.



Zinc, Zinc on Steel

Sectioning

Al₂O₃ Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	200	35
320	30	200	35
600	30	200	35

Polishing

3 micron diamond compound/
silk cloth/microid extender

60

200

35

1 micron diamond compound/
red felt cloth/microid extender

60

200

35

Optional:

0.05 micron gamma alumina/
Lecloth/alcohol

30

150

20

Remarks

Water will attack zinc coating. Use ethyl alcohol instead of water as lubricant for alumina polish.

Etchants

4% Picral

Zirconium

(Zr-2, Zr-4)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
120	60	300	40
320	60	300	40
400	60	300	40
600	60	300	40

Polishing

0.3 micron Al_2O_3 /nylon/
water and 10% Cr_2O_3

120

500

40

0.05 micron Al_2O_3 /Lecloth/water
or

240

150

20

colloidal silica/wetted Imperial Cloth

120

150

15

Remarks

To facilitate polishing, a solution composed of 75 ml H_2O , 4 ml HNO_3 and 10 drops HF can be added to the final polishing step by using an eyedropper. Examine under polarized light.

Etchants

30 ml Lactic acid, 30 ml HNO_3 , 10 ml HF
Swab

Note:

When etchant is first applied to sample, the sample will turn black. Continued swabbing will chemically polish and remove polishing scratches. Let etchant remain on sample without swabbing to reveal microstructure.



Zirconium Oxide—Metal Laminate

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Diallyl Phthalate, Epoxide

Grinding

	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
30–45 micron diamond grinding disc/water	120	300	40
10–20 micron diamond grinding disc/water	120	300	40

Polishing

9 micron diamond compound/ silk cloth/microid extender	240	250	35
1 micron diamond compound/ silk cloth/microid extender	120	250	35

Zirconium Oxide (Zr_2O_3) on Nickel or Steel Substrate

(Plasma Coating)

Sectioning

SiC Cutoff Wheel/Coolant

Mounting

Bakelite, Epoxide, or Castable Mounting Media

Grinding

SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	40
320	30	300	40
400	30	300	40
600	30	300	40

Polishing

3 micron diamond compound/ silk cloth/microid extender	300	250	35
1 micron diamond compound/ red felt cloth/microid extender	180	250	35
colloidal silica/Lecloth/water	60	150	30



Helpful Hints

- The silicon carbide grinding discs used with these procedures are PSA (Pressure Sensitive Adhesive Back). Plain back discs cannot be used because the samples go off the periphery of the wheel and through the center. (AP-60 and SS-1000 applications only).
- When possible, PSA cloths are recommended, but not absolutely necessary.
- When using the overhang type of cloths, wet thoroughly with water before attaching the retaining band; this helps to keep the cloths taut over the polishing wheels. This is recommended with the red felt cloth for diamond polishing also. Remove excess water by turning on polishing wheel at high speed.
- When using acids on polishing wheels, place a plastic barrier (Saran Wrap, e.g.) between the cloth and polishing wheel to eliminate a galvanic cell being established.
- It is not necessary to ultrasonically clean between grinding steps, but necessary after grinding and between intermediate and final polishing steps.
- Carbon steel samples should be rinsed and dried as soon as possible to avoid corrosion attack.
- Coplanarity between the sample surfaces and the specimen holder is essential before going to a succeeding step. Coplanarity is established during the first grinding step.
- Record deviations from listed parameters so reproducible results can be obtained in the future.
- If processing unmounted specimens, make sure all burrs are removed before loading into the specimen holder.
- Section off severe non-parallel surfaces of specimens that are to be processed in the unmounted condition; if non-parallelism is not too severe, grinding on a belt grinder will establish relative parallelism.
- Placing a shim on the center portion of the specimen leveler will allow specimens to protrude farther from the surface of the specimen holder.
- Etch-polishing ferrous alloys can be accomplished on specimens while still in the specimen holder by swabbing lightly with a cotton ball saturated in 2% Nital.
- Lower pressure is required when processing specimens having large metal surface areas; e.g., unmounted specimens.
- Do not continue to grind samples for the sake of getting one sample flat—drop it out and put it with another group.
- Do not remove samples from holder until the desired quality of surface finish is obtained. Use a microscope with an inverted stage for periodic microscopic examination.
- Coarser grits of silicon carbide grinding discs (120 and 180 grit) will process several holders of specimens; however, only one disc of the finer grades should be used per specimen holder.
- Avoid too much microid extender on the diamond polishing cloths; even with heavy pressure, the specimen holder can "hydroplane" over the surface.
- If comet tails are observed when processing specimens containing carbide phases, decrease the pressure and extend the polishing time.
- When preparing more samples than what one specimen holder can accommodate, use another holder and process both holders through the various grinding and polishing steps. Do not process one holder through the stages completely, then come back to the other holder. Time can be saved, especially since several holders can be processed through the coarser grinds.
- Exercise good housekeeping habits.
- Do not be hesitant about experimenting.

Composition of Etchants

2% Nital

100 ml Ethyl Alcohol
2 ml Nitric Acid

4% Picral

4 g Picric Acid
100 ml Ethyl Alcohol

10% Ammonium Persulfate

10 g Ammonium Persulfate
100 ml water

10% Oxalic Acid

10 g Oxalic Acid
100 ml water

10% Potassium Cyanide

10 g Potassium Cyanide
100 ml water

Barkers Reagent

2 to 4% Hydrofluorboric Acid
200 ml water

Chromic Acid

10 g Chromium Trioxide (Red Crystals)
100 ml water

Copper Etch

5 ml Sulfuric Acid
2 mm Hydrochloric
95 mm H₂O
10 ml of 10% Chromic Acid

Glyceregia

30 ml Glycerine
30 ml Hydrochloric
10 ml Nitric

Halls Reagent

20 g Potassium Permanganate
20 g Sodium Carbonate
20 g Sodium Hydroxide
8 g Potassium Dichromate
200 ml water

Kellers Reagent

94 ml water
3 ml Nitric Acid
2 ml Hydrochloric Acid
1 ml Hydrofluoric Acid

Krolls Reagent

94 ml water
4 ml HNO₃
2 ml HF

Marbles Reagent

4 g Copper Sulfate
20 ml water
20 ml Hydrochloric

Murakamis Reagent

4 g Potassium Ferricyanide
10 g Potassium Hydroxide
100 ml water

NOTE: 7 g Sodium Hydroxide may
be substituted for KOH

Vilella's Reagent

100 ml ethanol o-methanol
5 ml Hcl
1 g Picric Acid

Helpful Hints

- Wear gloves and eye protection when mixing etchants.
- Always pour the strong into the weak.
- When adding sulfuric acid to water, tip the container and allow sulfuric to run down the side.
- 20 drops (eyedropper) equals 1 ml.





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