

# *Automatic Polishers*

*Suggested Procedures for Grinding & Polishing Various Alloys*



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EMPOWERING RESULTS

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# 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.

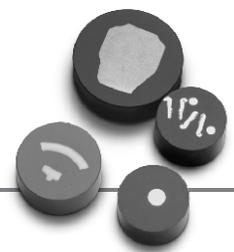
After polishing wheels have been initially charged with polishing media and wetted with the appropriate lubricants, the frequency of dispersion should be approximately every 60 seconds for the microid extender and every 30 seconds for the alumina slurry.

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](http://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

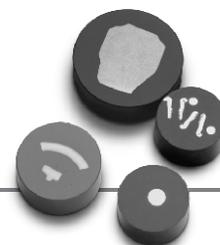
Bakelite or Castable Mounting Media

## 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

Al<sub>2</sub>O<sub>3</sub> 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 H<sub>2</sub>O, 1 part HCl, 1 part HF  
15% Aqueous NaOH (Immerse)

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# 2090 Aluminum with Li-Cu-Zr

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

<b>SiC Grit Size</b>	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

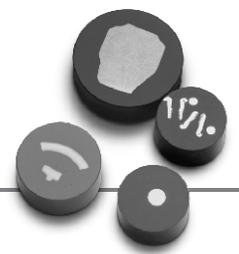
60

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

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# Aluminum, As-Cast

## Sectioning

Bandsaw, Al<sub>2</sub>O<sub>3</sub> 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

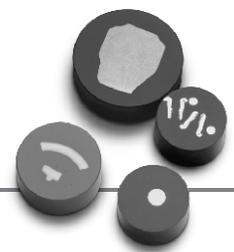
## Etchants

Barkers

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

Kellers

Immerse



# Aluminum, As-Cast

## Sectioning

Bandsaw, Al<sub>2</sub>O<sub>3</sub> 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

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# Aluminum, Unmounted Samples

(Unmounted Samples up to 1.5 inches in diameter.

Larger automatic polishers only.)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

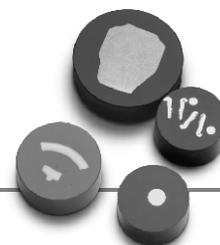
6 micron diamond compound/ red felt cloth/microid extender	240	250	30
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1 micron diamond compound/ red felt cloth/microid extender	240	250	30
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colloidal silica/Imperial Cloth/ wetted	180	100	0
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##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

Kellers



# Aluminum Oxide

(Ceramic)

## Sectioning

Diamond Cutoff Wheel

## Mounting

Castable Mounting Media. Avoid compression mounting unless samples are absolutely flat. Add Pelletized Al<sub>2</sub>O<sub>3</sub> (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.

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# Aluminum Oxide

(Sparkplug, Ceramic)

## Sectioning

Diamond Cutoff Wheel

## Mounting

Castable, add pelletized  $\text{Al}_2\text{O}_3$  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

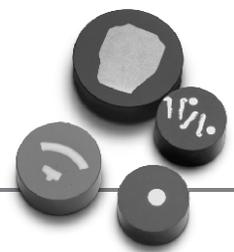
150

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**

Al<sub>2</sub>O<sub>3</sub> 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

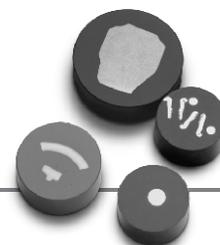
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## **Remarks**

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 <sup>(a)</sup>	120	300	40
400	60	300	40
600	60	300	40

<sup>(a)</sup>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.

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# Beryllium

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> 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

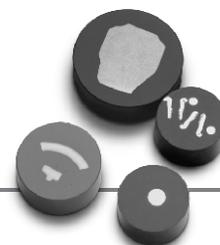
120

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

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# Brass

Using CAMEO® Magnetic Discs

## Sectioning

SiC or Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## Mounting

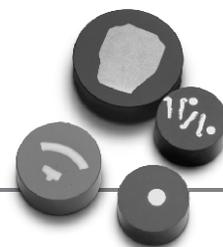
Bakelite, Epoxide, or Castable Mounting Media

## 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

Al<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub>/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

Al<sub>2</sub>O<sub>3</sub> 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

Al<sub>2</sub>O<sub>3</sub> 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

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# Cast Iron

Using CAMEO® Magnetic Discs

## Sectioning

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## Mounting

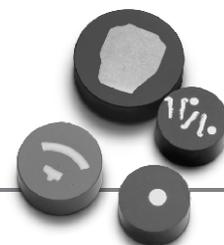
Bakelite, Epoxide, or Castable Mounting Media

## 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<sub>2</sub>O<sub>3</sub> 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 Al<sub>2</sub>O<sub>3</sub> 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

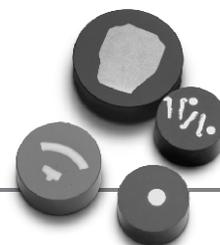
3 micron diamond compound/ silk cloth/microid extender	180	250	30
1 micron diamond compound/ red felt cloth/microid extender	60	250	30
colloidal silica/wetted Imperial Cloth	60	150	15

## 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

Al<sub>2</sub>O<sub>3</sub> 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 <sup>(a)</sup> /wetted Lecloth	60	100	20

<sup>(a)</sup>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

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# Ceramic

## **Sectioning**

Diamond Low-Deformation Saw

## **Mounting**

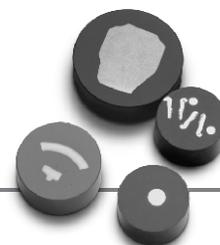
Castable Mounting Media

## **Grinding**

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (psi)</b>	<b>Wheel Speed (RPM)</b>
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

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# Coal

## **Sectioning**

Diamond Low-Deformation Saw

## **Mounting**

Castable Mounting Media

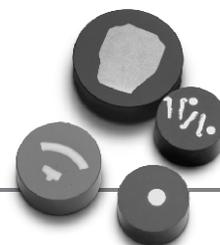
## **Grinding**

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (psi)</b>	<b>Wheel Speed (RPM)</b>
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
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0.05 micron colloidal silica/Imperial Cloth	45	CCW	75	35	150
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# Coal and Coke

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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 Al<sub>2</sub>O<sub>3</sub>/Lecloth/  
water/10% Cr<sub>2</sub>O<sub>3</sub><sup>(a)</sup>

240

300

40

<sup>(a)</sup>Chromic Acid—100 ml H<sub>2</sub>O, 10g Cr<sub>2</sub>O<sub>3</sub>

## 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<sup>®</sup> Magnetic Discs

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

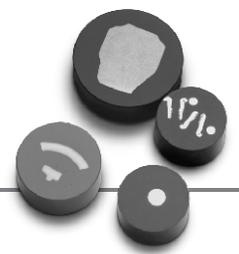
3:00	CCW	40	100	CCW	200
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1 micron premium  
suspension/red felt/  
microid extender

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**

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (psi)</b>	<b>Wheel Speed (RPM)</b>
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

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# Copper

Using CAMEO<sup>®</sup> Magnetic Discs

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

Al<sub>2</sub>O<sub>3</sub> 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 (0.05 $\mu$ ) can be used as the final polishing medium. The addition of a few drops of a solution composed of 50 ml NH<sub>4</sub>OH and 5 ml H<sub>2</sub>O<sub>2</sub> will facilitate polishing.

## Etchants

50 ml ammonium hydroxide (NH<sub>4</sub>OH), 5 ml hydrogen peroxide (30%) H<sub>2</sub>O<sub>2</sub>

Immerse

NOTE: If etchant is too fast, add 50 ml H<sub>2</sub>O.

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.

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# Copper Alloys

(Brasses)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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
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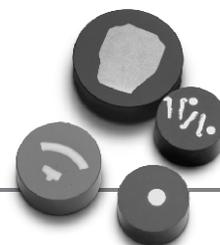
1 micron diamond compound/red felt cloth/ microid extender	180	250	35
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0.05 micron gamma alumina/Lecloth/water <sup>(a)</sup>	180	150	40
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<sup>(a)</sup>Optional—To keep lead clean and metallic looking.  
Polishing may terminate with 1 micron diamond polish.

## Etchants

50 ml H<sub>2</sub>O, 50 ml NA<sub>4</sub>OH, 5 ml H<sub>2</sub>O<sub>2</sub>  
Immerse



# Copper Alloys

(Bronzes)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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
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colloidal silica/ wetted Imperial Cloth	120	150	15
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## Etchants

1 g NaCl, 95 ml H<sub>2</sub>O, 2 ml 20% chromic acid, 2 ml H<sub>2</sub>SO<sub>4</sub>  
Immerse

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

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# Copper–Beryllium Alloys

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

Ferric Oxide + 10% Cr<sub>2</sub>O<sub>3</sub><sup>(a)</sup>/  
Lecloth

120

150

30

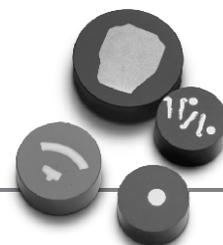
<sup>(a)</sup> 10% Cr<sub>2</sub>O<sub>3</sub> = 100 ml H<sub>2</sub>O, 10 g Cr<sub>2</sub>O<sub>3</sub>

## 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
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1 micron diamond compound/ silk over silk/microid extender	300	200	30
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0.05 micron Al <sub>2</sub> O <sub>3</sub> /Lecloth/ water	300 to 600	100	30
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## 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<sub>4</sub>OH, 5 ml H<sub>2</sub>O<sub>2</sub>  
Immerse

If etching too fast, add 50 ml H<sub>2</sub>O

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# Glass

## Sectioning

SiC Cutoff Wheel/Coolant

## Mounting

Castable Mounting Media

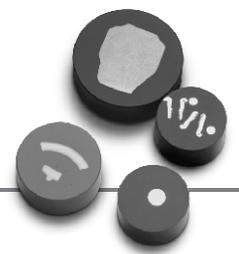
## Grinding

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 <sup>(a)</sup> /wetted Lecloth	120	150	15

<sup>(a)</sup>Rare earth oxides in suspension



# Glass

## **Sectioning**

Diamond Low-Deformation Saw

## **Mounting**

Unmounted or Castable Mounting Media

## **Grinding**

	<b>Time (sec.)</b>	<b>Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# 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

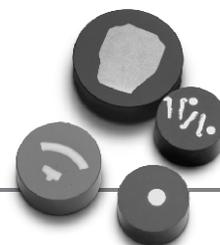
## Polishing

3 micron premium  
diamond suspension/  
ultra silk/  
microid extender

6:00 CW 35 100 CCW 200

0.05 micron colloidal  
silica/imperial cloth

2:00 CW 25 75 CCW 150



# 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

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# Incoloy/Inconel

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

1 micron diamond compound/  
red felt cloth/microid extender

60

250

30

0.05 micron gamma alumina/  
wetted Lecloth

or

colloidal silica/wetted

Imperial Cloth

60

250

30

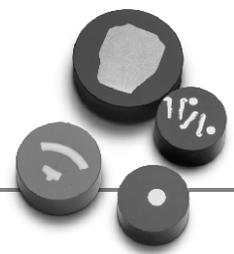
## Remarks

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 HNO<sub>3</sub>  
Swab



# Inconel

Using CAMEO® Magnetic Discs

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

Al<sub>2</sub>O<sub>3</sub> 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

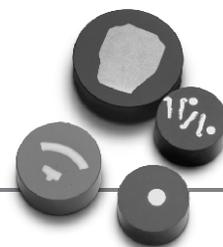
## Remarks

Polishing can terminate with the diamond polish.

## Etchants

2% Nital

4% Picral



# Lead

## Sectioning

Microtome Best.  $\text{Al}_2\text{O}_3$  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
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colloidal silica/ wetted Imperial Cloth	180	100	—
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## Remarks

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

## Etchants

60 ml acetic acid, 20 ml  $\text{H}_2\text{O}_2$ , 1 ml HCl  
Immerse

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

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# Magnesium Casting

## **Sectioning**

SiC Cutoff Wheel/Coolant

## **Mounting**

Cold Mount, Acrylic, or Bakelite

## **Grinding**

	<b>Time (min:sec)</b>	<b>Head Direction</b>	<b>Head Pressure (lb.)</b>	<b>Head Speed (RPM)</b>	<b>Wheel Direction</b>	<b>Wheel Speed (RPM)</b>
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**

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

<b>SiC Grit Size</b>	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# Nickel Alloys

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

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

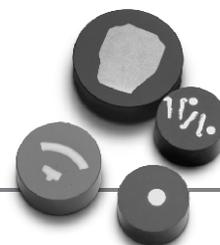
6 micron diamond compound/ silk cloth/microid extender	300	250	32
6 micron diamond compound/ red felt cloth/microid extender	180	250	32
3 micron diamond compound/ silk cloth/microid extender	300	250	32
1 micron diamond compound/ red felt cloth/microid extender	120	250	32
colloidal silica/Imperial Cloth/ 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 HNO<sub>3</sub>, Electrolytic, 0.8 A, 2 sec.



# Nickel-Base Superalloys

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

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

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# Nickel-Base Superalloys

(Turbine Blades, etc.)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

Al<sub>2</sub>O<sub>3</sub> 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 Al<sub>2</sub>O<sub>3</sub>/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 HNO<sub>3</sub>, 5 ml HF  
Swab

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# Plasma Spray

(WC, CrC, Cr<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, Zr<sub>2</sub>O<sub>3</sub>, Al-Si, etc. Coatings on Ni, Steel, Inconel Substrates)

## Sectioning

SiC Cutoff Wheel/Coolant

## Mounting

Epoxide (Compression), Fluorescent Castable

## Grinding

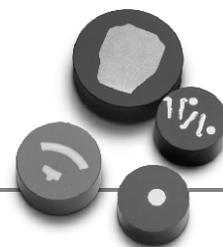
SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	120	300	35
240	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

## 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.

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# Plastic

## Sectioning

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel

## Mounting

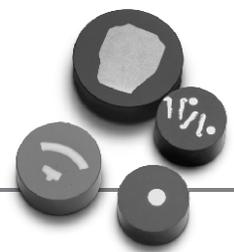
Castable Mounting Media (low heat)

## Grinding

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (psi)</b>	<b>Wheel Speed (RPM)</b>
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

1 micron premium  
suspension/red felt/  
microid extender

0:30 CW 30 100 CCW 200

0.05 micron colloidal  
silica/imperial cloth

0:30 CW 30 75 CCW 150

## 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.

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# Rene

## **Sectioning**

SiC Abrasive Cutoff Wheel

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (psi)</b>	<b>Wheel Speed (RPM)</b>
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

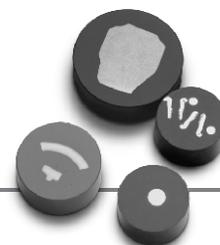
## **Polishing**

9 micron diamond compound/  
red felt cloth/  
microid extender

240 CCW 75 20 150

0.05 micron colloidal  
silica/Imperial Cloth

120 CCW 75 20 150



# Resulfurized Steel

(11XX, 12XX Alloys)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

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

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> 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<sub>2</sub>O<sub>3</sub>/Lecloth

60

100

35

## **Remarks**

Ferric Oxide Slurry: 500 ml H<sub>2</sub>O, 20 grams ferric oxide, 15 ml 10% Cr<sub>2</sub>O<sub>3</sub>, 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 H<sub>2</sub>O, 10 ml HCl, 10 ml H<sub>2</sub>O<sub>2</sub>  
Immerse

100 ml H<sub>2</sub>O, 50 g sodium hydroxide

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# Silicon Carbide

## **Sectioning**

Diamond Cutoff Wheel/Coolant

## **Mounting**

Castable Mounting Media

## **Grinding**

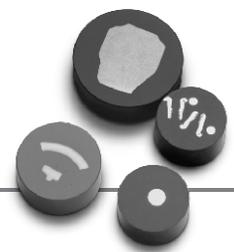
	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
63–74 micron diamond grinding disc/water	180	200	30
30–40 micron diamond grinding disc/water	180	200	30
10–20 micron diamond grinding disc/water	60	200	30

## **Polishing**

6 micron diamond compound/ silk cloth/microid extender	240	200	30
1 micron diamond compound/ red felt cloth/microid extender	60	200	30
<i>Optional:</i> 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**

	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# Silicon Carbide on Graphite

## **Sectioning**

SiC Cutoff Wheel/Coolant

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

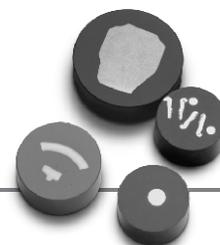
	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
63–74 micron diamond grinding disc/water	120	300	35
10–20 micron diamond grinding disc/water	180	300	35

## **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

Al<sub>2</sub>O<sub>3</sub> 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

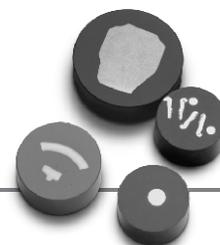
1:00	CCW	10	100	CCW	200
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0.05 micron colloidal  
silica/imperial cloth

4:00	CCW	10	75	CCW	150
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## Etchant

20 ml NH<sub>4</sub>OH 20 ml H<sub>2</sub>O<sub>2</sub>, 10 ml H<sub>2</sub>O; swab for 3 to 10 seconds.



# Silver with Cadmium Oxide

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

<b>Sic Grit Size</b>	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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 H<sub>2</sub>O, 25 ml NH<sub>4</sub>OH, 3 ml H<sub>2</sub>O<sub>2</sub>  
Immerse

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# Stainless Steel

(Austenitic)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

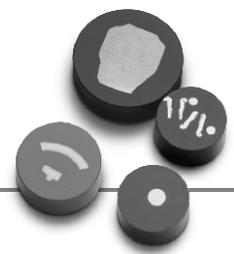
1 micron diamond compound/ red felt cloth/microid extender	240	250	30
colloidal silica/ wetted Imperial Cloth	120	150	15

## Etchants

30 ml Glycerine, 30 ml HCl, 10 ml HNO<sub>3</sub>  
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

Al<sub>2</sub>O<sub>3</sub> 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

Al<sub>2</sub>O<sub>3</sub> 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

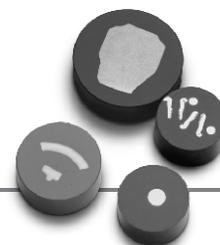
60

CW

75

45

150



# Steel

Using CAMEO® Magnetic Discs (HRB < 100)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

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# Steel

(Low to Medium Carbon)

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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 compound/ 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 H<sub>2</sub>O, 5 ml H<sub>2</sub>SO<sub>4</sub> and 5 ml H<sub>2</sub>O<sub>2</sub>.



# Steel

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

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## **Mounting**

Bakelite, Epoxide, or Castable Mounting Media

## **Grinding**

<b>SiC Grit Size</b>	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# Tantalum

(TA Alloys)

## Sectioning

SiC Cutoff Wheel/Coolant

## Mounting

Bakelite, Epoxide, or Castable Mounting Media

## Grinding

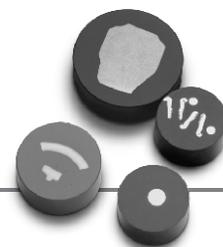
SiC Grit Size	Time (sec.)	Wheel Speed (RPM)	Pressure (psi)
180	60	300	35
320	60	300	35
400	60	300	35
600	60	300	35

## 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 HNO<sub>3</sub>, 5 ml HF  
Swab



# Tin

## **Sectioning**

Al<sub>2</sub>O<sub>3</sub> Cutoff Wheel/Coolant

## **Mounting**

Castable Mounting Media

## **Grinding**

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

## **Polishing**

0.05 micron Al<sub>2</sub>O<sub>3</sub>/Lecloth/  
water + NH<sub>4</sub>OH & H<sub>2</sub>O<sub>2</sub>

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% H<sub>2</sub>SO<sub>4</sub> in 100 ml H<sub>2</sub>O, electrolytic, 1A, 5 sec.

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

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# Titanium

Using CAMEO<sup>®</sup> Magnetic Discs

## Sectioning

Al<sub>2</sub>O<sub>3</sub> 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

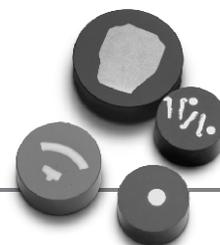
90

CCW

75

35

150



# Titanium 6-4

Using CAMEO® Magnetic Discs

## Sectioning

SiC Cutoff Wheel/Coolant

## Mounting

Bakelite, Epoxide, or Diallyl Phthalate

## Grinding

	<b>Time (sec.)</b>	<b>Direction</b>	<b>Head Speed (RPM)</b>	<b>Pressure (lb.)</b>	<b>Wheel Speed (RPM)</b>
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

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# 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

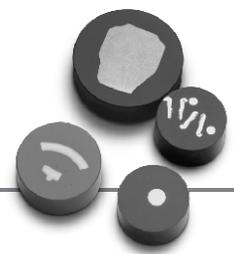
## Remarks

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 HNO<sub>3</sub>, 2 ml HF



# Titanium Alloy with SiC Inserts

## **Sectioning**

Diamond Cutoff Wheel/Coolant

## **Mounting**

Diallyl Phthalate

## **Grinding**

	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# Tungsten Carbide

Using CAMEO® Magnetic Discs

## Sectioning

Diamond Cutoff Wheel/Coolant

## Mounting

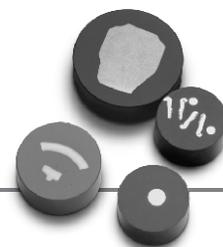
Epoxide or Diallyl Phthalate (glass-filled)

## 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**

	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# 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 <sub>2</sub> O <sub>3</sub> /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**

Al<sub>2</sub>O<sub>3</sub> 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

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# 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 <sub>2</sub> O <sub>3</sub> /nylon/ water and 10% Cr <sub>2</sub> O <sub>3</sub>	120	500	40
0.05 micron Al <sub>2</sub> O <sub>3</sub> /Lecloth/water or colloidal silica/wetted Imperial Cloth	240 120	150 150	20 15

## Remarks

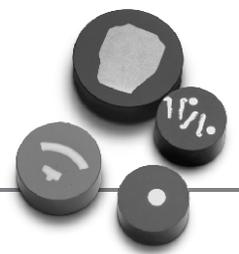
To facilitate polishing, a solution composed of 75 ml H<sub>2</sub>O, 4 ml HNO<sub>3</sub> and 10 drops HF can be added to the final polishing step by using an eyedropper. Examine under polarized light.

## Etchants

30 ml Lactic acid, 30 ml HNO<sub>3</sub>, 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**

	<b>Time (sec.)</b>	<b>Wheel Speed (RPM)</b>	<b>Pressure (psi)</b>
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

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# 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

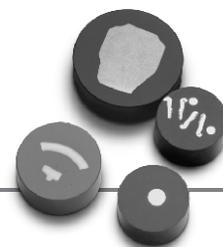
35

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.

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# 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 ml Hydrochloric  
95 ml H<sub>2</sub>O  
10 ml of 10% Chromic Acid

## **Glyceredia**

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 HNO<sub>3</sub>  
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

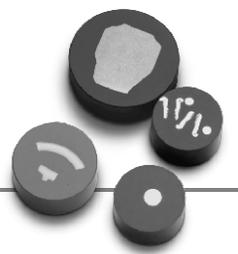
NOTE: 7 g Sodium Hydroxide may  
be substituted for KOH

## **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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