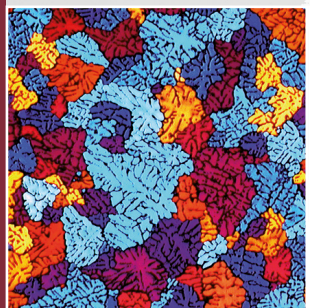
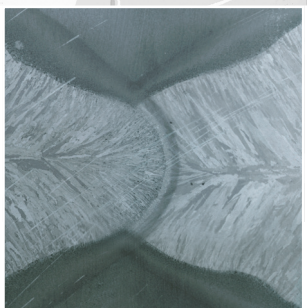
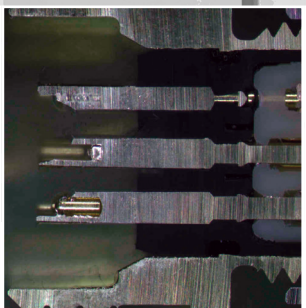
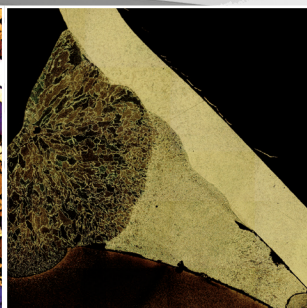
ADVANCED MATERIALOGRAPHY

Sample preparation

Standard preparation methods for materialographic analysis

Practical experience ATM-Lab



ADVANCED MATERIALOGRAPHY

NOTES:
The preparation methods can be applied for proper sectioned and burred specimens. Parameters like pressure (given as force in newton) are referred to single pressure and depend on specimen size.
For specimens ≠ 40mm these values have to be adjusted with the help of table „Pressure parameters and specimen size".
Instead of the Dia Complete diamond suspensions common diamond suspensions in combination with lubricants (water or alcohol based) can be used. Mind right dosage!
The velocity of the sample holder is 100 rpm and can vary depending on the specific material.
Due to the diversity of materials and examination aims the provided standard preparation methods might not cover all applications. In this case contact our application lab; our team will support you with customized preparation methods.

lab@atm-m.com

Grit conversion chart

FEPA (P)	grain size [µm]	ANSI/CAMI	grain size [µm]
60	269	60	268
80	201	80	188
100	162	100	148
120	127	120	116
180	787	180	78
240	58.5	220	66
280	52.2	240	51.8
320	46.2		
360	40.5	280	42.3
400	35	320	34.3
500	30.2	360	27.3
600	25.8	400	22.1
800	21.8		
1000	18.3	500	18.2
1200	15.3	600	14.5
1500	12.6	800	12.2
2000	10.3	1000	9.2
2500	8.4	1200	6.5
4000	5		

Reference:
Analytical Characterization of Aluminum, Steel and Superalloys
D. Scott MacKenzie, George E. Totten
October 10, 2005 by CRC Press

ISBN: 9780824758431

Pressure parameters and specimen size

Specimen diameter [mm]	25	30	40	50	60
Divergence in pressure used in the preparation methods	- (5N…10N)	- 5N	0	+ 5N	+ (5N…10N)

NOTES:
The preparation methods can be applied for proper sectioned and burred specimen. Parameters like pressure (given as force in newton) are referred to single pressure and depend on specimen size. For specimens ≠ 40mm these values have to be adjusted.

EXPLANATION OF SYMBOLS

Symbol	Meaning
	Cutting
	Mounting
	Grinding / Polishing
	Grinding / Polishing
	Etching
	Pre-polishing
	Polishing / diamond
	Final polishing

Symbol	Meaning
	Single pressure
	Speed grinding disc
	Speed sample holder
▶▶	Sample holder clockwise
◀▶	Sample holder anti-clockwise
	Time
	Dosing lubricant
	Lubricant waterbased
	Lubricant alcohol based

MATERIAL — Aluminum (≥99,7%) and wrought aluminum alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Bakelite red/black
KEM 20, KEM 30

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil** P320 (280)	H_2O	250-300	►►	20	Until plane
Grinding	SiC-paper/foil** P600 (400)	H_2O	250-300	►►	20	1:00
Grinding	SiC-paper/foil** P1200 (600)	H_2O	250-300	►►	20	1:30 (change SiC paper/foil after 45sec)
Polishing	GAMMA	Dia Complete Poly, 3µm	120-150	►►	30	6:00
Final polishing	LAMBDA/OMEGA	Eposil F 0.1µm	120-150	◄ ►	20	2:00 (H_2O for last 30sec)
Etching (electrolyt.)	Barker's reagent					30V

** To reduce the contamination of the specimen by SiC particles, it should be coated with paraffin wax before grinding

MATERIAL Aluminum alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Bakelite black/red
KEM 20

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	25	Until plane
Pre-polishing	BETA	Dia Complete Poly, 9µm	120-150	◄ ►	25	5:00
Polishing	SIGMA	Dia Complete Poly, 3µm	120-150	►►	30	5:00
Final polishing	LAMBDA/OMEGA	Eposil F 0.1µm	120-150	◄ ►	20	2:00 (H_2O for last 30sec)
Etching (chem.)	Kroll's reagent					0:30

MATERIAL Carbon/glass fiber reinforced composites (CFC/GFC)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: diamond, metal bond (bronze)
Anti-corrosion coolant

MOUNTING

Equipment
Pressure vessel

Consumable
KEM 15 plus

Method
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	35	Until plane
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	30	5:00
Polishing	GAMMA/DELTA	Dia Complete Poly, 3µm	120-150	►►	30	5:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	1:30 (H_2O for last 30sec)

MATERIAL Cast iron (GJS/GJL)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Bakelite red/black
KEM 30

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	Until plane
Grinding	SiC-paper/foil P600 (400)	H_2O	250-300	►►	30	1:00
Grinding	SiC-paper/foil P1200 (600)	H_2O	250-300	►►	30	1:00
Polishing	SIGMA	Dia Complete Poly, 3µm	120-150	►►	25	5:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄►	20	1:00 (H_2O for last 10sec)*
Etching (chem.)	Nital					0:03-0:10

* Rinsing with water can cause corrosion

MATERIAL — Soft to medium-hard steel (<35 HRC/350HV)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black, Bakelite red/black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	Until plane
Pre-polishing	BETA	Dia Complete Poly, 9µm	120-150	◄ ►	35	5:00
Polishing	SIGMA/GAMMA	Dia Complete Poly, 3µm	120-150	►►	30	6:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	1:00 (H_2O for last 30sec)
Etching (chem.)	Nital (micro) Adler's reagent (macro)*					0:03 0:03

* For weld analysis

MATERIAL — Medium-hard to hard steel (35-65 HRC/850HV)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	GALAXY red	H_2O	250-300	►►	30	Until plane
Pre-polishing	BETA	Dia Complete Poly, 9µm	120-150	◄ ►	30	5:00
Final polishing	IOTA	Dia Complete Poly, 3µm	120-150	◄ ►	30	4:00

MATERIAL Stainless steel (austenitic/ferritic)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black, Bakelite red/black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	Until plane
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	25	5:00
Polishing	GAMMA	Dia Complete Poly, 3µm	120-150	►►	25	5:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	2:00 (H_2O for last 30sec)
Etching (chem.)	V2A reagent					0:05-0:30

MATERIAL — Steel and welded steel (macro)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black, Bakelite red/black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P180 (180)	H_2O	250-300	►►	30	Until plane
Grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	35	1:00
Etching (chem.)	Adler's reagent (macro)					0:03-0:10

MATERIAL Nitrided steel

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black**
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	GALAXY green	H_2O	250-300	►►	25	Until plane
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	25	5:00
Polishing	GAMMA/DELTA	Dia Complete Poly, 3µm	120-150	►►	30	5:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	15	1:00 (H_2O for last 30sec)

** Before hot mounting the specimen should be wrapped in aluminum foil to stabilize the nitrided layer

MATERIAL Ceramics

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: diamond, metal bond (bronze)
Anti-corrosion coolant

MOUNTING

Equipment
Vacuum

Consumable
KEM 90 (porous material)
KEM 35 (high-density, solid material)

Method
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	GALAXY red	H_2O	250-300	►►	30	Until plane
Grinding	GALAXY blue	H_2O	250-300	►►	30	2:00
Pre-polishing	BETA	Dia Complete Poly, 9µm + diamond paste	120-150	◄ ►	40	6:00
Final polishing	GAMMA	Dia Complete Poly, 3µm + diamond paste	120-150	►►	30	5:00

MATERIAL Cobalt based alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin or rubber bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	Until plane
Pre-polishing	BETA	Dia Complete Poly, 9µm	120-150	◄ ►	30	8:00
Polishing	GAMMA	Dia Complete Poly, 3µm	120-150	►►	30	6:00
Polishing	ZETA/IOTA	Dia Complete Poly, 1µm	120-150	►►	30	3:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	2:00 (H_2O for last 30sec)

MATERIAL — Nickel based alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin or rubber bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black
KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	GALAXY green	H_2O	250-300	►►	30	Until plane
Pre-polishing	BETA	Dia Complete Poly, 9µm	120-150	◄ ►	30	6:00
Polishing	GAMMA	Dia Complete Poly, 3µm	120-150	►►	30	5:00
Final polishing	LAMBDA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	1:30-2:00 (H_2O for last 30sec)

MATERIAL Spray coatings (metallic, ceramic)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: diamond, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Vacuum

Consumable
KEM 90

Method
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P180 (180)	H_2O	250-300	►►	30	Until plane
Grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	1:30
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄►	30	5:00-8:00**
Polishing	GAMMA	Dia Complete Poly, 3µm	120-150	►►	30	5:00-8:00**
Polishing	ZETA	Dia Complete Poly, 1µm	120-150	►►	20	2:00
Final polishing	LAMBDA	Eposal 0.06µm	120-150	◄►	20	1:30 (H_2O for last 30sec)

** Until constant porousity ➔ next polishing step

MATERIAL Magnesium

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: diamond, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Bakelite red/black, Thermoplast, KEM 20

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil* P600 (400)	H_2O	250-300	►►	15	Until plane
Polishing	BETA	Diamond suspension (alcohol or oil based) 9µm, poly	120-150	►►	15	5:00
Polishing	SIGMA	Diamond suspension (alcohol or oil based) 3µm, poly	120-150	►►	15	5:00
Polishing	ZETA	Diamond suspension (alcohol or oil based) 1µm, poly	120-150	►►	15	5:00
Final polishing	OMEGA**	Etosil E 0.06µm	120-150	◄►	15	4:00 (ethanol for last 30sec)
Etching (chem.)	3% nitric acid					0:03-0:10 (ethanol for 30sec)

* To reduce the contamination of the specimen by SiC particles, it should be coated with paraffin wax before grinding

** Wet the polishing cloth with ethanol before polishing

MATERIAL — Printed circuit board (non assembled)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Pressure vessel

Consumable
KEM 20

Method
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P180 (180)	H_2O	250-300	►►	30	Until plane (slightly before point of interest)
Grinding	SiC-paper/foil P800 (500)	H_2O	250-300	►►	25	1:00 (until point of interest)
Grinding	SiC-paper/foil P1200 (600)	H_2O	250-300	►►	25	1:00 (until point of interest)
Polishing	GAMMA/DELTA	Dia Complete Poly, 3µm	120-150	►►	30	3:00
Final polishing	ZETA/LAMBDA	Eposal 0.06µm	120-150	◄►	25	2:00 (LAMBDA: H_2O for last 30sec)

MATERIAL — Printed circuit board (assembled)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: diamond, metal bond
Anti-corrosion coolant

MOUNTING

Equipment
Vacuum
Pressure vessel

Consumable
KEM 90, KEM 20

Method
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280), GALAXY green**	H_2O	250-300	►►	30	Until plane
Grinding	SiC-paper/foil P600 (400), GALAXY blue**	H_2O	250-300	►►	30	1:00
Grinding	SiC-paper/foil P1200 (600), GALAXY yellow**	H_2O	250-300	►►	30	1:00
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	25	4:00
Polishing	GAMMA/DELTA	Dia Complete Poly, 3µm	120-150	►►	25	4:00
Final polishing	ZETA/OMEGA	Eposal 0.06µm	120-150	◄ ►	20	1:30 (LAMBDA: H_2O for last 30sec)

** For printed circuit boards with ceramic components

MATERIAL Copper and copper alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Bakelite red/black, Thermoplast
KEM 20, KEM 30

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	30	Until plane
Grinding	SiC-paper/foil** P600 (400)	H_2O	250-300	►►	30	1:30
Grinding	SiC-paper/foil** P1200 (600)	H_2O	250-300	►►	30	1:30
Polishing	SIGMA	Dia Complete Poly, 3µm	120-150	►►	30	4:00
Final polishing	OMEGA	Eposil F 0.1µm**	120-150	◄►	15	2:00 (H_2O for last 30sec)
Etching (chem.)	Klemm's I reagent					0:02

** 50ml Eposil F + 1ml H_2O_2 + 1ml NH_3

MATERIAL — Titanium (commercial pure: grade 1-4)

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black
KEM 20, KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	25	Until plane
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	20	10:00
Final polishing	LAMBDA	Eposil F 0.1µm**	120-150	◄ ►	20	10:00 – 15:00* (H_2O for last 30sec)
Etching (chem.)	Kroll's reagent					0:45

* Depends on grade of titanium

** Eposil F has to be mixed with hydrogen peroxide (35%) in a ratio of 5:1
(safety advice: use personal protective equipment)

MATERIAL Titanium alloy

CUTTING

Equipment
Brillant

Consumable
Cut-off wheel: corundum, resin bond
Anti-corrosion coolant

MOUNTING

Equipment
Opal

Consumable
Epo black
KEM 20, KEM 15 plus

Method
Hot mounting
Castable

GRINDING/ POLISHING

Specimen size Ø 40 mm

STEP	MEDIUM		rpm		N	min
Planar grinding	SiC-paper/foil P320 (280)	H_2O	250-300	►►	25	Until plane
Grinding	SiC-paper/foil P600 (400)	H_2O	250-300	►►	25	1:30
Pre-polishing	ALPHA/BETA	Dia Complete Poly, 9µm	120-150	◄ ►	20	5:00
Final polishing	LAMBDA	Eposil F 0.1µm**	120-150	◄ ►	20	5:00 – 10:00* (H_2O for last 30sec)
Etching (chem.)	Kroll's reagent					0:45

* Depends on the alloy

** Eposil F has to be mixed with hydrogen peroxide (35%) in a ratio of 5:1
(safety advice: use personal protective equipment)

ADVANCED MATERIALOGRAPHY

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