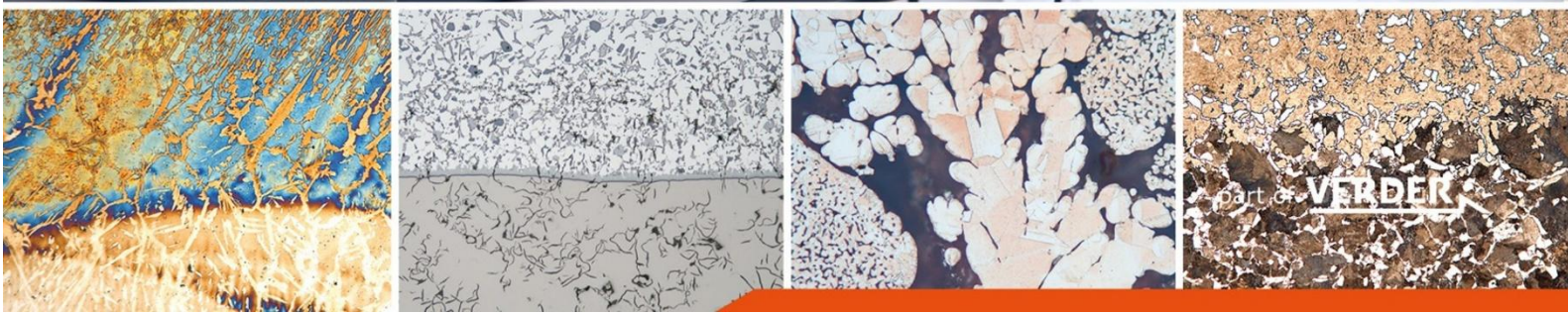


Application lab. Report

Preparation to analyze the
microstructure of the Zirconium



QATM-Preparation method

Objective:

The challenge with this task is to avoid using hazardous acids during the polishing process. And the preparation method for these zirconium samples should be appropriate for EBSD investigation and other investigations. Qpol Vibro was then used to see the difference.

We have sensitive, thin samples and standard samples. The sensitive samples require a two-step method for hot mounting.

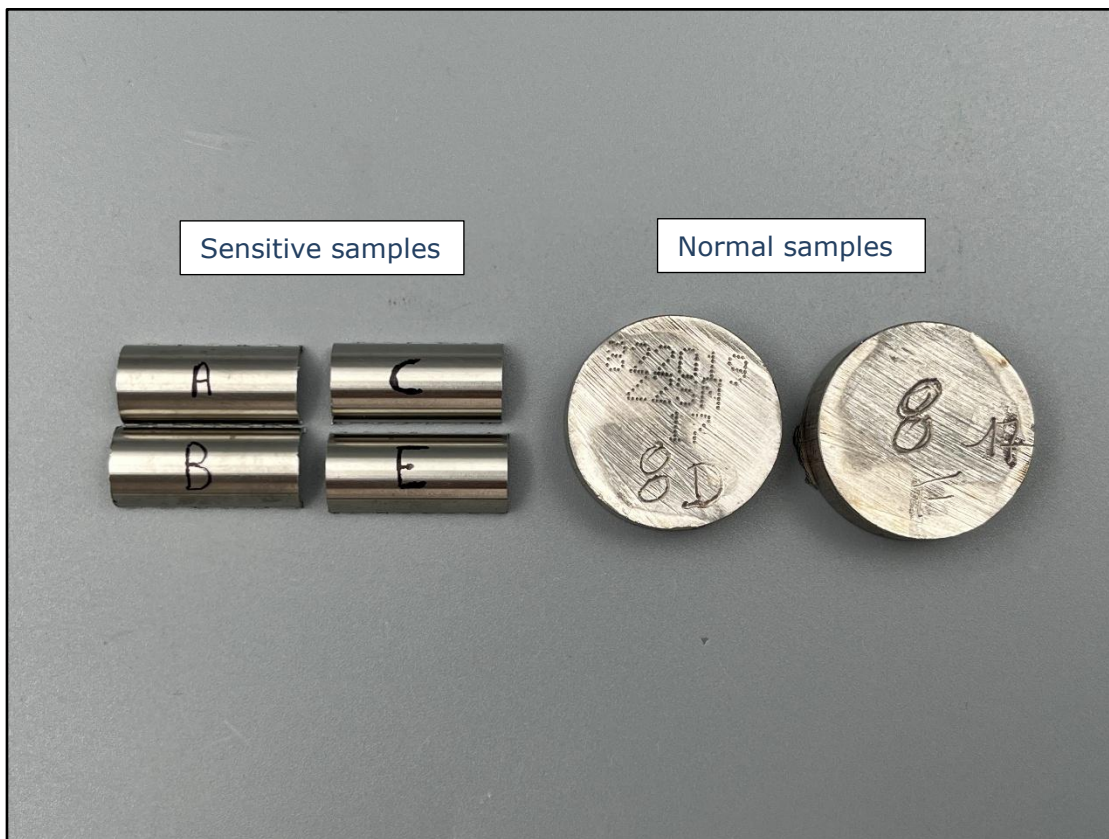


Figure 1. As-received samples

QATM-Preparation method

Hot mounting

 Mounting					
Device	Consumable	Heating time	Temperature	Pressure	Cooling time
Qpress 40	DUROPLAST BLACK	3:00 min 4:00 min	120 °C 200 °C	10 bar 180 bar	3:30 min
Filler or additional consumables	Heating power	Pressure mode	Cooling power		
-	100%	2-Step Modus	100%		
Notes					
These parameters are only for the sensitive samples in two Steps modus and for ø30 mm!					



Figure 2. Overview of the samples after mounting

QATM-Preparation method

Grinding/Polishing

Device	Samples holder	Pressure mode					
QPOL Plus 1	Z5446022	Single ø30mm					
Step	MEDIUM		 RPM		 N	 min	
 Planar grinding	Self-adhesive SiC-Paper P320 + X-Tap	H ₂ O	200	120 ▶▶	25	1:00 Till flat	
 Grinding	Self-adhesive SiC-Paper P600 + X-Tap	H ₂ O	200	120 ▶▶	25	1:00	
 Pre-Polishing	BETA	Dia Complete Poly, 9µm	150	120 ◀▶	30	6:00	
 Fine polishing	OMEGA	Eposil F 0,1µm +H ₂ O ₂	90	100 ◀▶	30	15:00 (Rinsing time : 30 s)	
 Primary etching (chem.)	Etchant Kroll					0:05	
 Fine polishing	OMEGA	Eposil F 0,1µm +H ₂ O ₂	90	100 ◀▶	30	15:00 (Rinsing time : 30 s)	
 Finally etching (chem.)	Etchant Kroll					0:05-0:10	

Notes

- Pre-dosing for 9 µm: 3 s
Dosing interval and dosing duration for Dia. Suspension 9 µm:
Every 30 s for 1,3 s
- Dosing interval and Dosing duration for fine polishing suspension:
Every 20 s for 1,3 s

QATM-Preparation method

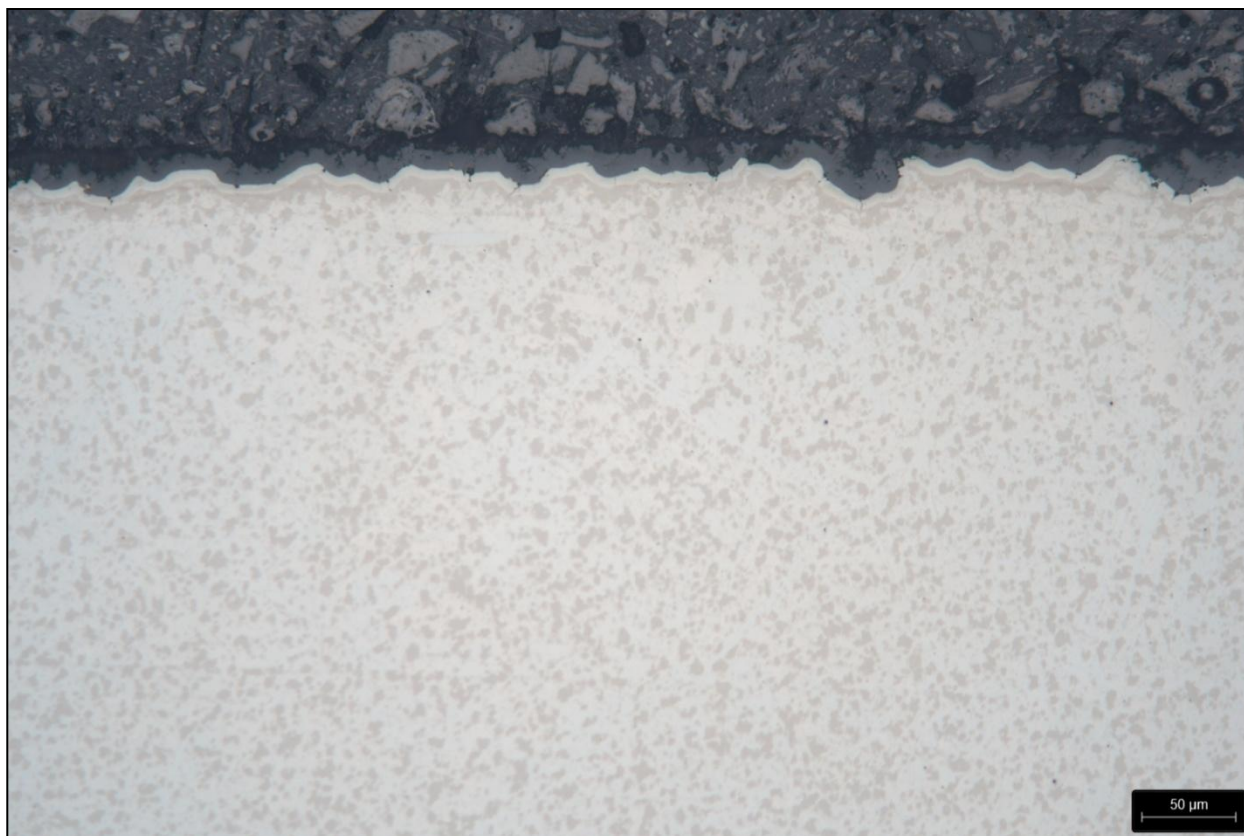


Figure 3. Sample 8F after fine-polishing with Eposil F and without primary etching – 200 : 1



Figure 4. Sample 8F after fine-polishing with Eposil F and etched with Kroll – 200 : 1

QATM-Preparation method



Figure 5. Sample 8F after fine-polishing with Eposil F and without primary etching – 200 : 1



Figure 6. Sample 8F after fine-polishing with Eposil F and etched with Kroll – 200 : 1

QATM-Preparation method

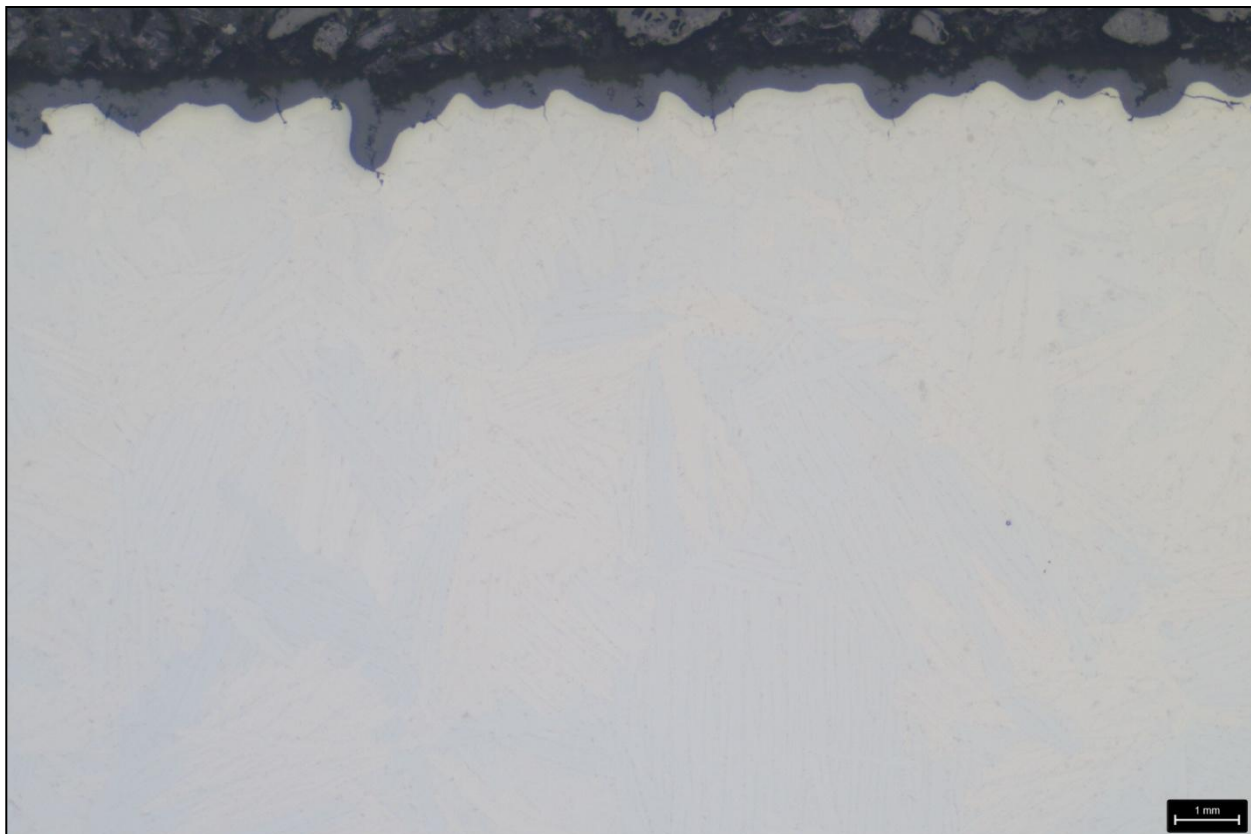


Figure 7. Sample 8F after fine-polishing with Eposil F and primary etching – 200 : 1

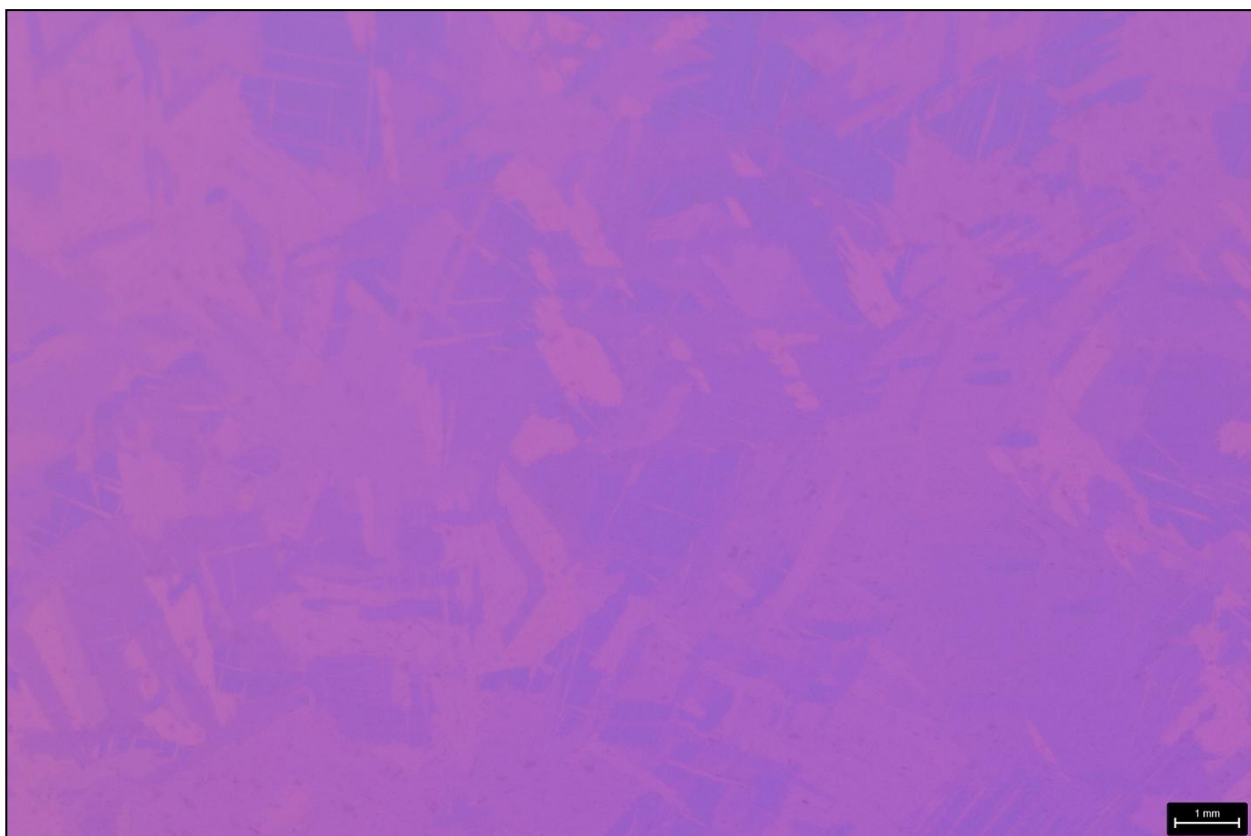


Figure 8. Sample 8F after fine-polishing and primary etching- polarized light – 200 : 1

QATM-Preparation method



Figure 9. Sample A after fine-polishing with Eposil F without primary etching – 200 : 1



Figure 10. Sample A after fine-polishing and primary etching- polarized light – 200 : 1

QATM-Preparation method

Vibration polishing

Device	Sample holder	Weight	
QPOL VIBRO	Ø 30 mm	3 × 90 g	
Step	MEDIUM		Frequency (Hz)  min

	Vibration-Polishing	IOTA	Eposil F	60	120
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Notes

- In addition to the frequency of 60 Hz, an intensity of 70% should be set



Figure 11. Overview of the levelling plate

QATM-Preparation method – after Qpol Vibro

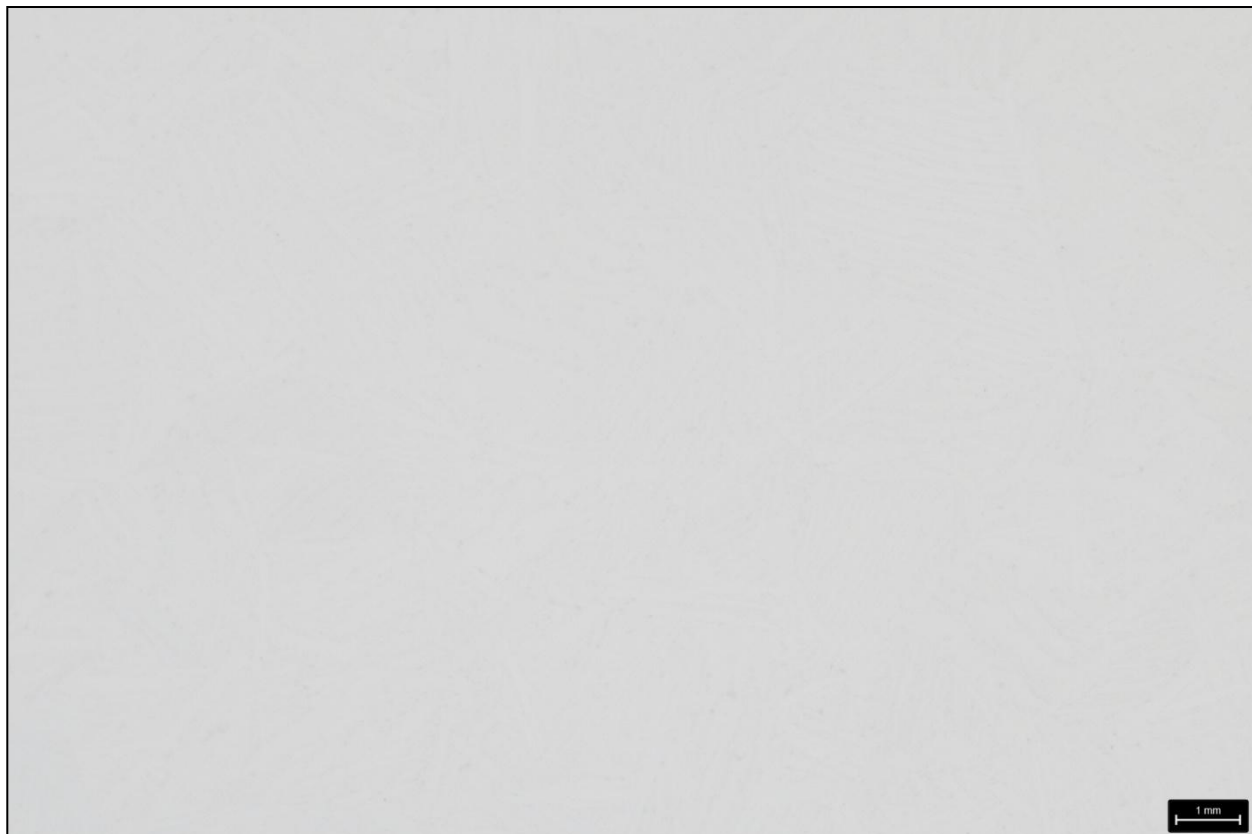


Figure 12. Sample 8F after Qpol Vibro for 2 hour and primary etching – 200 : 1

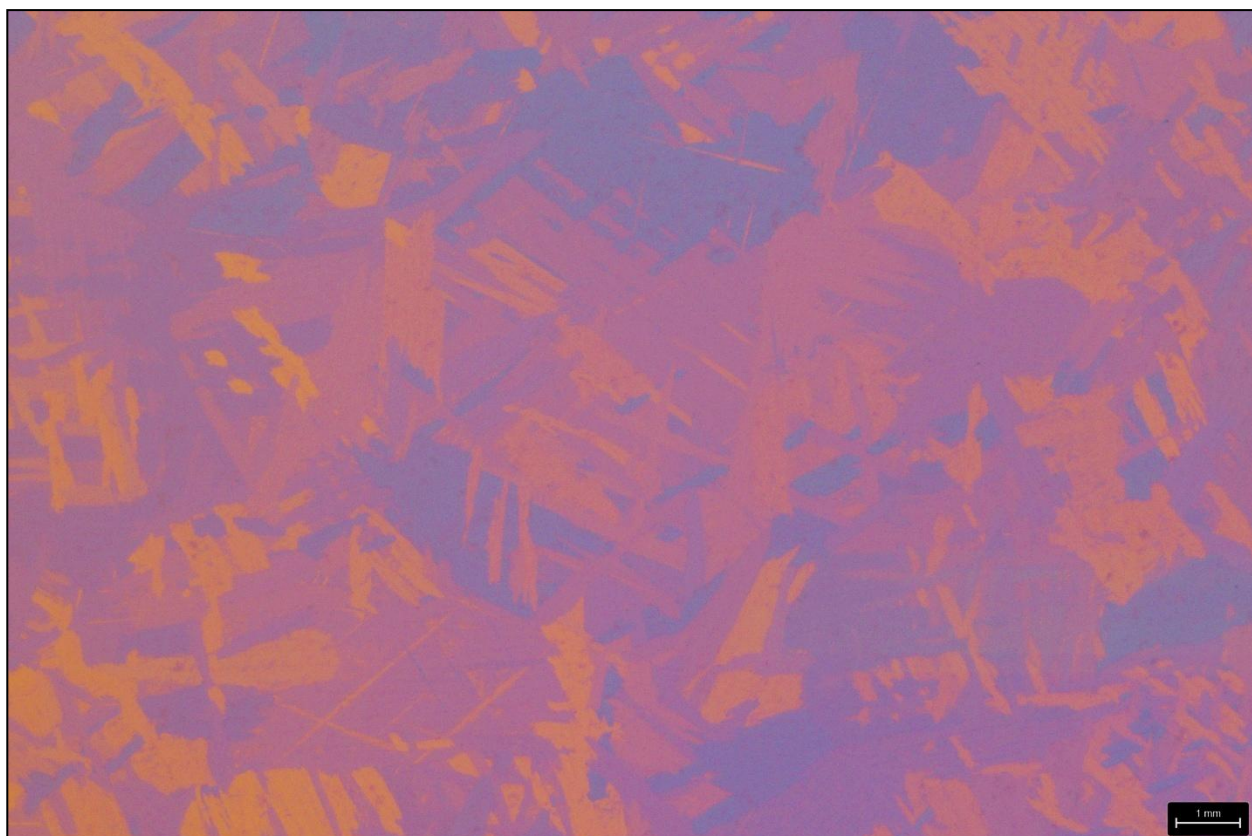


Figure 13. Sample 8F after Qpol Vibro and primary etching- polarized light – 200 : 1

QATM- Preparation method

Consumables and accessories used:

	Consumables and accessories	Article description	Item number
Grinding	Sample holder	Sample holder M	Z5446022
	Fixation system	X-Tap	95017021
	Grinding paper	Self-adhesive SiC-Paper P320	92001659
	Grinding paper	Self-adhesive SiC-Paper P600	92001661
	Diamond suspension	Dia-Complete poly. 9µm	95011843
	Polishing cloth	BETA	95001412
	Polishing cloth	OMEGA, ø 300mm (fine polish)	95005781
	Polishing cloth	IOTA, ø 300mm (Vibro)	95001421
	Fine polishing suspension	Eposil F, 0,1µm	92002536
Etching	Etchant	Kroll	92004492

Conclusion:

1. The 2 step-method for hot mounting is well suited to sensitive, fragile components. The other samples can be mounted as normal at full pressure.
2. We recommend using our Galaxy X-Tap fixation system for grinding. This allows for quick replacement of self-adhesive grindpaper without leaving any adhesive residue, and you'll achieve a finish with a very sharp edge.
3. To avoid the use of hydrofluoric acid, we recommend briefly immersing the samples in Kroll after the first fine-polishing step and polishing them again after cleaning. We have also mixed Eposil F in a proportion of 1:5 with hydrogen peroxide to intensify the polishing effect. Immediately afterwards, the microstructure is reasonably clear to see with polarized light. The option to etch it again at the end is still available.
4. Befor use the Qpol Vibro we recommend using the levelling plate to clamp the sample so that it is exactly parallel (see Figure 11).