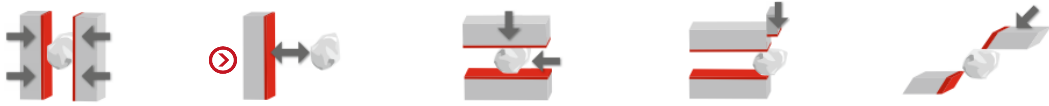

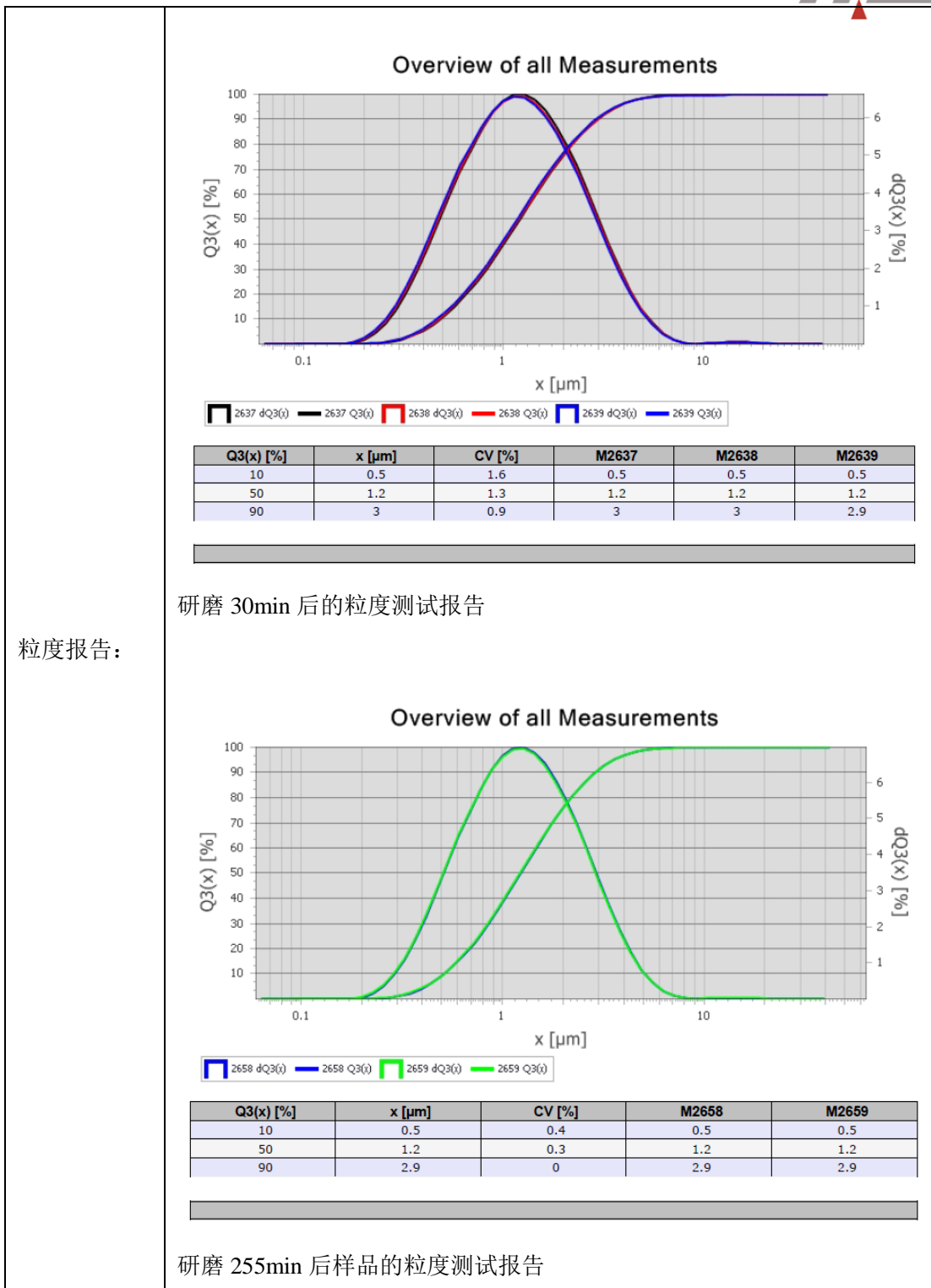


实验报告

实验编号:

日期: 2017.11.17

样品名称:	氧化铝	所属领域:	材料
原始尺寸:	微米级	期望细度:	100nm、200nm
样品量:	3g	后续分析:	电镜
其他要求:			
			
解决方案:			
所选机型:	微型行星式球磨机 Pulverisette 7 加强型		
配置:	80ml 氧化锆研磨罐 + 60g 1mm 氧化锆研磨球		
转速:	800rpm、1000rpm、1100rpm		
分散剂:	水		
研磨时间:	255min		
最终细度:	D50<1.2 μ m		
实验说明:	<p>1、称量加入 60g 氧化锆研磨球, 3g 样品, 水 30ml</p> <p>2、采用 800rpm 转速, 研磨 3min 暂停 7min 为一个周期, 循环 10 个周期。总研磨时间为 30min。</p> <p>3、继续研磨, 探究细度能否继续降低, 设置研磨转速为 1000rpm, 研磨 3min 暂停 12min 为一个周期, 循环 55 个周期, 总研磨时间为 195min。</p> <p>4、再次取样测量, 粒度没有明显变化。</p> <p>5、将转速提高至 1100rpm, 采用研磨 3min 暂停 8min 的周期, 研磨 20 个循环。取样测量粒度, 依旧没有明显变化。</p> <p>6、如果想要进一步降低样品粒度, 可以换用更小的研磨球 (针对 Al₂O₃ 样品, 建议换用 100g0.1mm 氧化锆研磨球), 增加转速及研磨时间很难使样品粒度进一步减小。</p> <p>7、借鉴德国总部应用工程师的经验, 对于 Al₂O₃ 粉体湿磨的方式可以将样品粒度减小至 D50<50nm。</p>		



ALUMINIUM OXIDE

Protocol number:	M110155
Industry:	Chemistry, Mechanochemistry
Feed Size:	< 70µm
Desired Fineness:	d50 < 50nm

Result 1



PLANETARY MICRO MILL PULVERISETTE 7 PREMIUM LINE

(speed: 1100 rpm)

80 ml grinding bowl made of zirconium oxide (ZrO₂) + 100g 0,5 mm ZrO₂ balls

Feed quantity:	4,8 g (5 ml)
Feed Size:	< 70 µm
Additive:	15ml H ₂ O*1
Grinding time:	60 min*2
Final fineness:	< 6 µm*3
Comments:	*1: For grinding the sample, we had to add 15ml of water (H ₂ O).

*2: For avoiding overpressure by grinding in suspension, we grinded in steps of 3 minutes, followed by 8 minutes of programmed pausing time. This counts for all further grinding trials too. If the outside temperature of the bowl will become too high (>70 °C), grinding/ pausing times should be readjusted.

*3: After 60min, the balls can be changed to 0,1mm diameter balls (see trial 1b).

Result 2



PLANETARY MICRO MILL PULVERISETTE 7 PREMIUM LINE

(speed: 1100 rpm)

80 ml grinding bowl made of zirconium oxide (ZrO₂) + 100g
0,1 mm ZrO₂ balls

Feed quantity: 4,8 g (~5 ml)
Feed Size: < 6 µm*1
Additive: +25ml water*2 (sum: 40ml)
Grinding time: + 4 hours*3 (sum: 5 h)
Final fineness: d50 < 50 nm*4
Comments: *1: After 1 hour with 0,5mm balls, particles are too fine for further grinding success with 0,5mm balls. Therefore, we had to change the balls to 0,1mm.

*2: To maintain an oil-like viscosity, we had to add 25ml of water additionally. Totally, 40ml of water have been added to the sample.

*3: After a grinding time of totally 5 hours, 50% (d50) of the sample used to show a diameter of 50nm. For achieving a better fineness, a longer grinding time should still be possible.

*4: See our particle size distribution after 5 hours of grinding, measured with our ANALYSETTE 22 NanoTec plus, meas. no. 31613 on separate page.

Download: [AL203__2_5HOURS.PDF](#)

Mie Theorie

Corundum
 Brechungsindex n = 1.760
 Absorptionskoeffizient a = 0.000
 Water (20°C)
 Brechungsindex n = 1.3328

d[4,3] = .07µm		Arithm. Mittel = 0.065 µm		Spezifische Oberfläche = 2173789.75 cm ² /cm ³				
Interpolationswerte... C:\Fritsch\la22_32\fritsch\001-2µm.FPS								
4.0 %	<=	0.011 µm	27.9 %	<=	0.020 µm	45.4 %	<=	0.030 µm
57.0 %	<=	0.040 µm	65.4 %	<=	0.050 µm	71.7 %	<=	0.060 µm
80.3 %	<=	0.080 µm	85.8 %	<=	0.100 µm	89.5 %	<=	0.120 µm
93.1 %	<=	0.150 µm	95.3 %	<=	0.180 µm	96.3 %	<=	0.200 µm
98.5 %	<=	0.300 µm	99.0 %	<=	0.400 µm	99.2 %	<=	0.500 µm
99.2 %	<=	0.600 µm	99.3 %	<=	0.800 µm	99.4 %	<=	1.000 µm
99.5 %	<=	1.200 µm	99.6 %	<=	1.500 µm	99.8 %	<=	2.000 µm

Interpolationswerte... C:\Fritsch\la22_32\fritsch\5_99.fpv								
5.0 %	<=	0.011 µm	10.0 %	<=	0.013 µm	15.0 %	<=	0.015 µm
20.0 %	<=	0.017 µm	25.0 %	<=	0.019 µm	30.0 %	<=	0.021 µm
35.0 %	<=	0.024 µm	40.0 %	<=	0.026 µm	45.0 %	<=	0.030 µm
50.0 %	<=	0.034 µm	55.0 %	<=	0.038 µm	60.0 %	<=	0.043 µm
65.0 %	<=	0.050 µm	70.0 %	<=	0.057 µm	75.0 %	<=	0.066 µm
80.0 %	<=	0.079 µm	85.0 %	<=	0.097 µm	90.0 %	<=	0.124 µm
95.0 %	<=	0.175 µm	98.0 %	<=	0.262 µm	99.0 %	<=	0.389 µm

