

SpiralTOF™

The Relationship between Crystal Condition and Mass Resolving Power, Mass Accuracy

Introduction

The JMS-S3000 SpiralTOF™ has a unique 17 m flight path that offers the highest resolution MALDI-TOF MS system currently available. With an extended flight distance, the SpiralTOF reduces topographic effect of matrix crystal to a minimum and achieves highly reproducible mass resolving power and high mass accuracy with external mass calibration.

In this work, we demonstrate the measurement of a polymer standard with 4 types of matrices that are typically used for MALDI polymer measurement by using the JEOL SpiralTOF system. Additionally, we looked at the crystal condition using the JEOL JSM-7600F thermal field emission scanning electron microscope (FE-SEM).

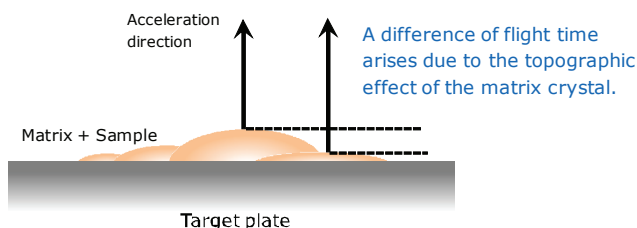


Figure 1. Reduced topographic effect of matrix crystal.

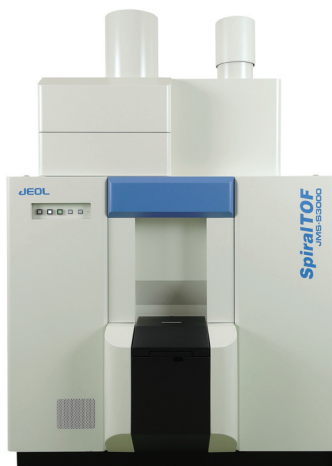


Figure 3. JMS-3000 SpiralTOF.

Experimental

Sample information and preparation conditions are shown in Table 1. PEG1500 was dissolved in water at a concentration of 10 mg/mL. Each matrix was dissolved in THF at a concentration of 10 mg/mL. NaI used as the cationization agent was dissolved in THF at a concentration of 1 mg/mL. Next, the PEG1500, NaI and matrix solutions were mixed together 1:1:2 (1:1:4 for DIT) by volume. Afterwards, 0.75 µL of this mixture was placed on the hairline finish stainless steel plate (MTP format, 384 spots for samples and 96 spot for calibrant). Finally, the dried sample was measured using the JMS-S3000 SpiralTOF MS system. We also obtained SEM images for each crystal condition with the JSM-7600F.

Polymer standard	Conc.	Solvent
Polyethylene glycol (PEG) 1500	10 mg/mL	Water
Cationization agent		
NaI	1 mg/mL	Water
Matrix		
α-Cyano-4-hydroxycinnamic acid (CHCA)	10 mg/mL	Tetrahydrofuran (THF)
2,5-Dihydroxybenzoic acid (DHB)	10 mg/mL	THF
Dithranol (DIT)*	10 mg/mL	THF
trans-3-Indoleacrylic acid (IAA)	10 mg/mL	THF
Sample		
PEG1500/NaI/Matrix = 1/1/2 (v/v)		
* PEG1500/NaI/DIT = 1/1/4 (v/v)		
0.75 µL of this sample solution mixture was placed on the MALDI target plate		
JSM-7600F conditions		
Sample preparation	Uncoated	
Acceleration voltage	1 kV	
Magnification	x500 and x2,000	

Table 1. Sample information and preparation conditions.



Figure 4. JSM-7600F Thermal FE-SEM.

Results & discussion:

The MALDI mass spectra of PEG1500 are shown in Figure 4 for each matrix. We set the delay time to achieve the maximum mass resolving power at m/z 1537.9. Therefore, the resolving power was approximately 70,000 for the $[\text{HO}(\text{C}_2\text{H}_4\text{O})_{34}\text{H} + \text{Na}]^+$ peaks, well in excess of that needed to separate isotope peaks. Additionally, we observed excellent mass distributions.

We determined the average mass resolving power ($n=10$) and external mass accuracy ($n=8$) for m/z 1097.6, m/z 1537.9 and m/z 1978.2 for each matrix. The results are shown in Figure 5 and 6, respectively. We achieved high mass resolving power for the three selected ions with each matrix. In addition, we obtained excellent mass error (less than 10 ppm) with external calibration for each matrix.

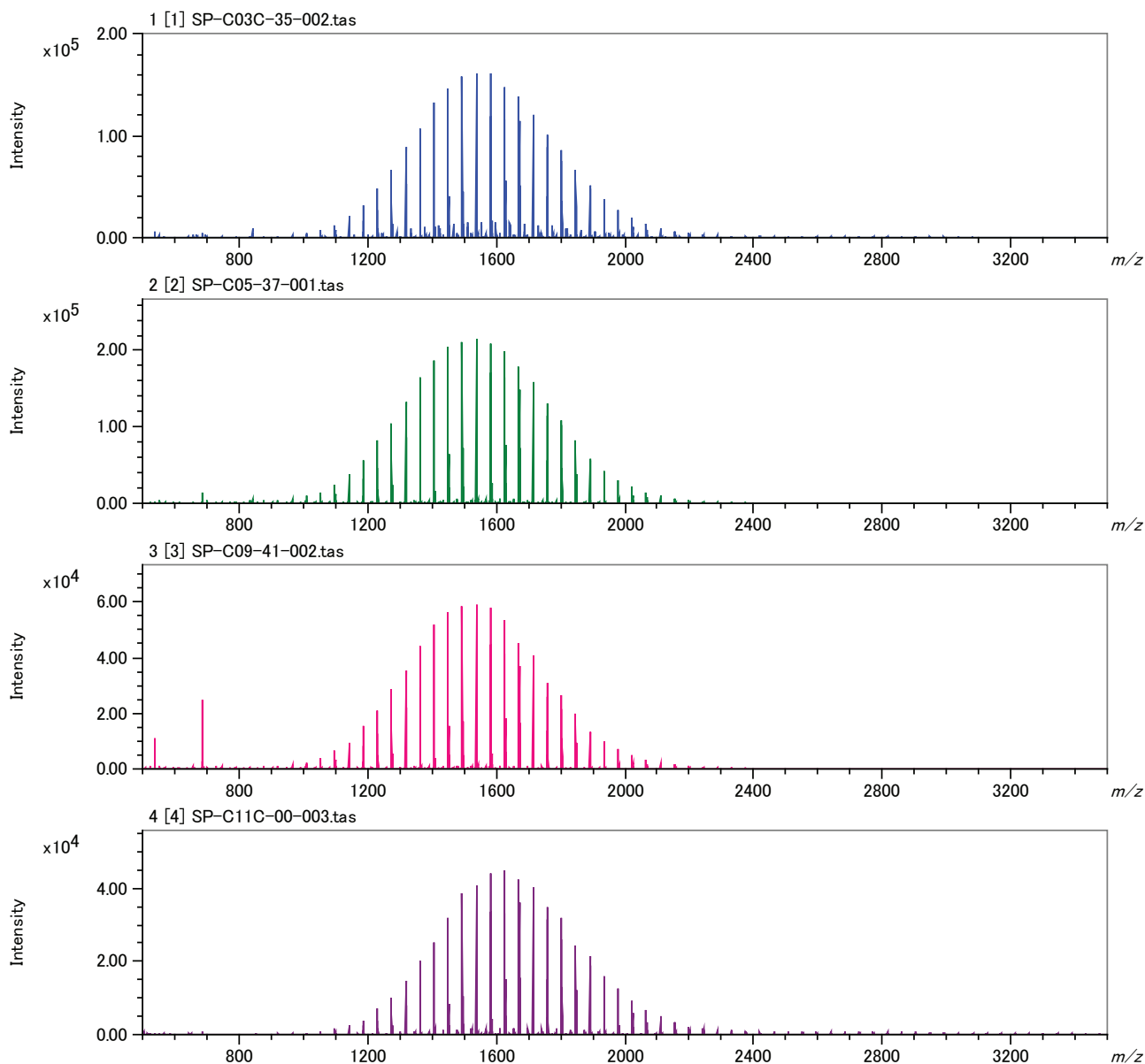


Figure 4. MALDI mass spectra of PEG1500.

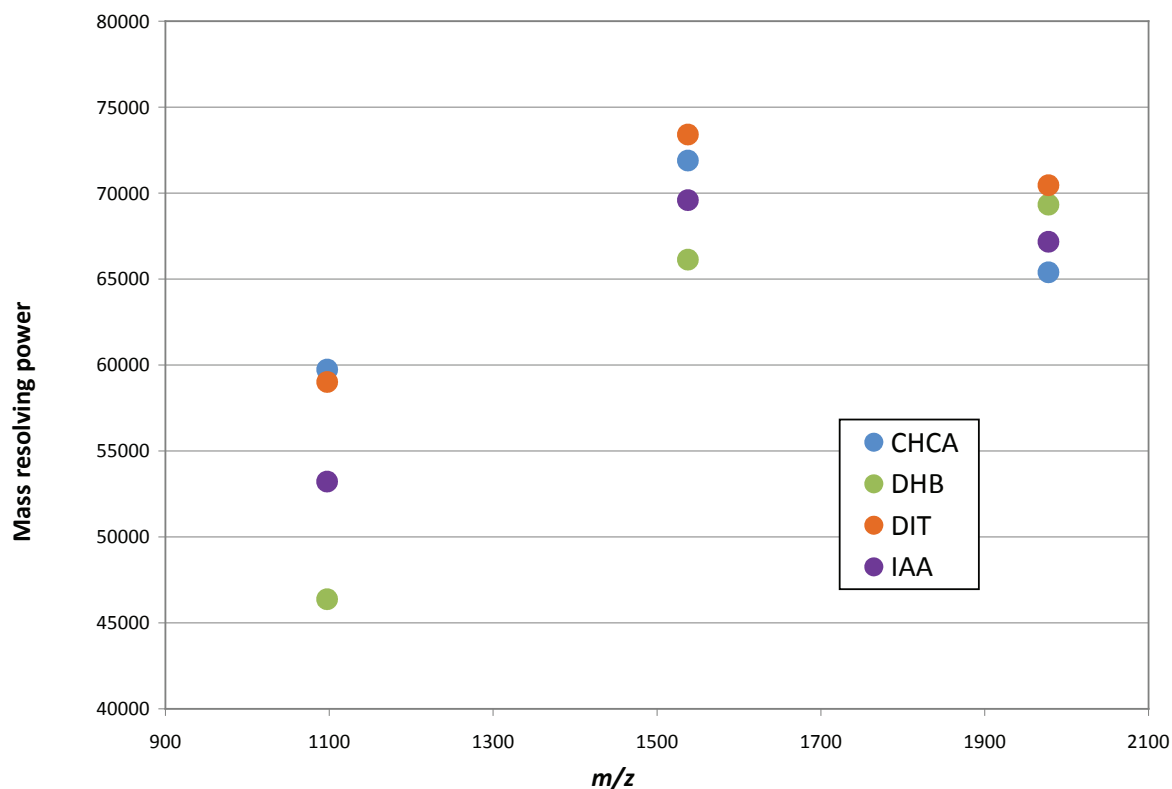


Figure 5. Averaged mass resolving power (n=10) for m/z 1097.6, m/z 1537.9 and m/z 1978.2.

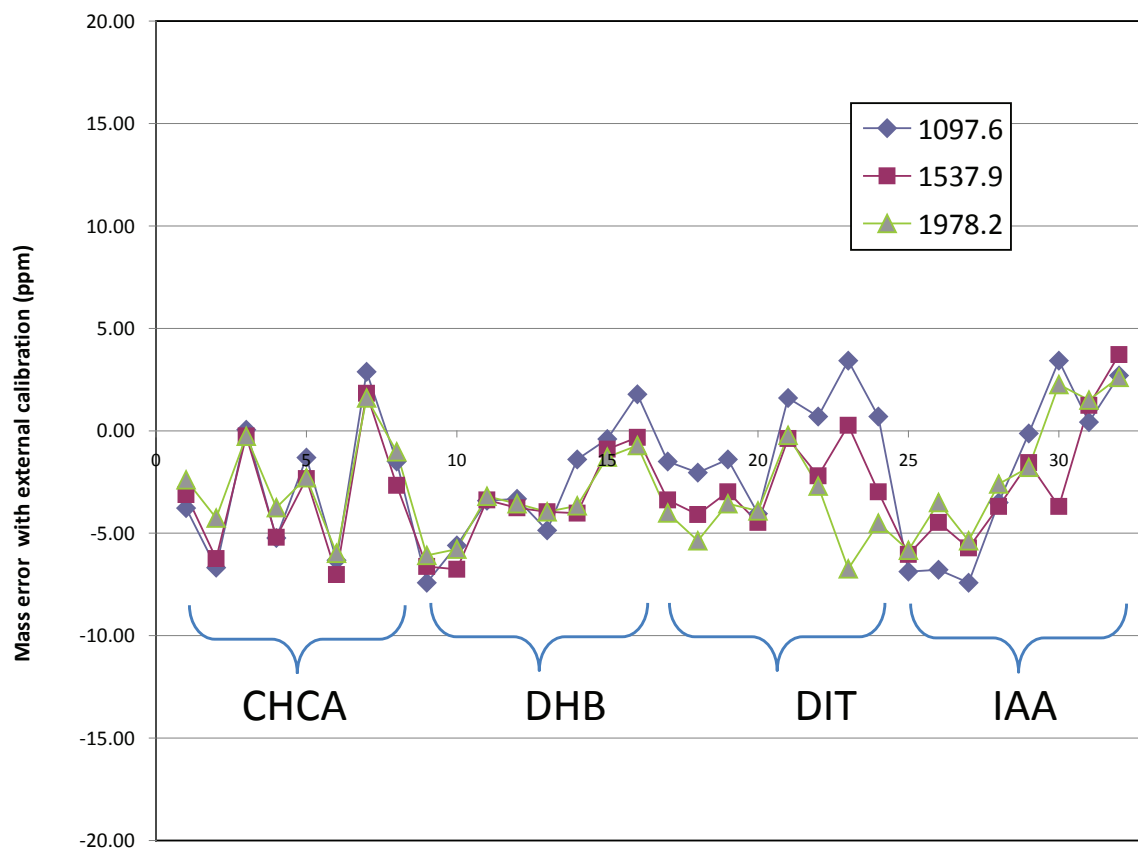


Figure 6. Mass error with external calibration (n=8) for m/z 1097.6, m/z 1537.9 and m/z 1978.2.

We examined the crystal condition with the JEOL JSM-7600F thermal field emission scanning electron microscope. The SEM images are shown Figures 7-10. The crystal shape, size and dispersion were quite different in each matrix crystal. However, SpiralTOF performance was not affected by the topographic effects because these spatial differences were a negligible fraction of the 17 m flight path.

Conclusions

SpiralTOF achieved highly reproducible mass resolving power and high mass accuracy with external mass calibration for all samples. These values were not significantly influenced by the different crystal morphologies for the different matrices. This is attributed to the SpiralTOF's very long (17 meter) flight path.

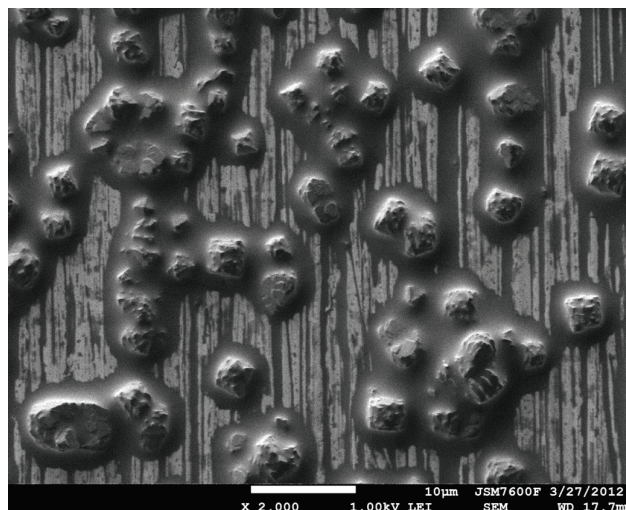
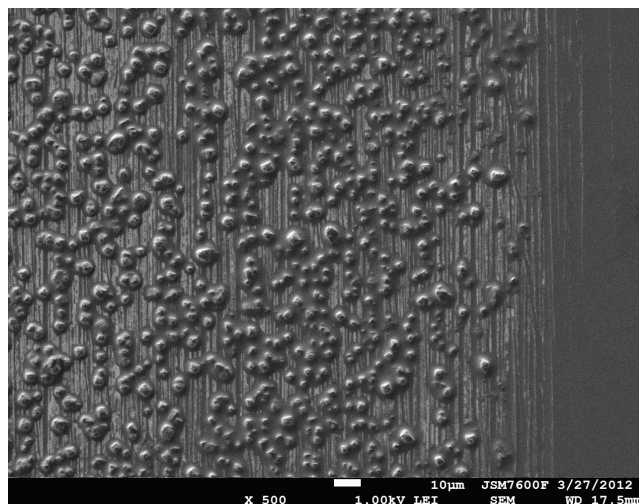


Figure 7. SEM images of CHCA crystal with PEG1500: left: x500, right: x2,000.

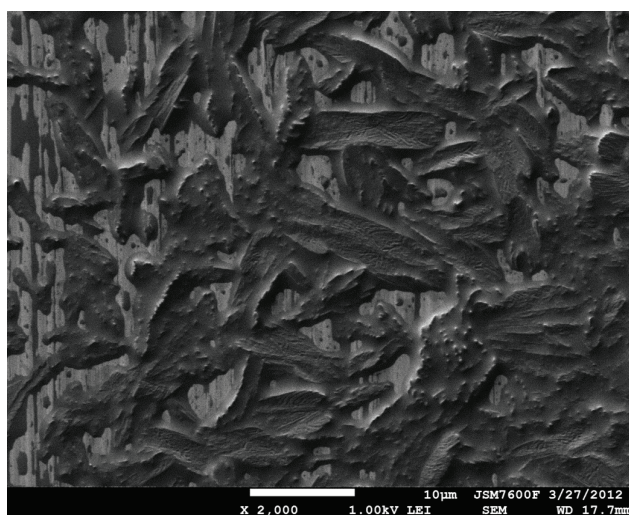
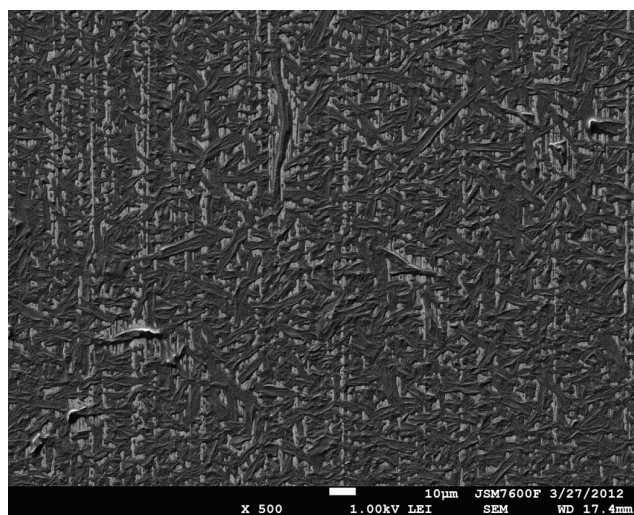


Figure 8. SEM images of DHB crystal with PEG1500: left: x500, right: x2,000.

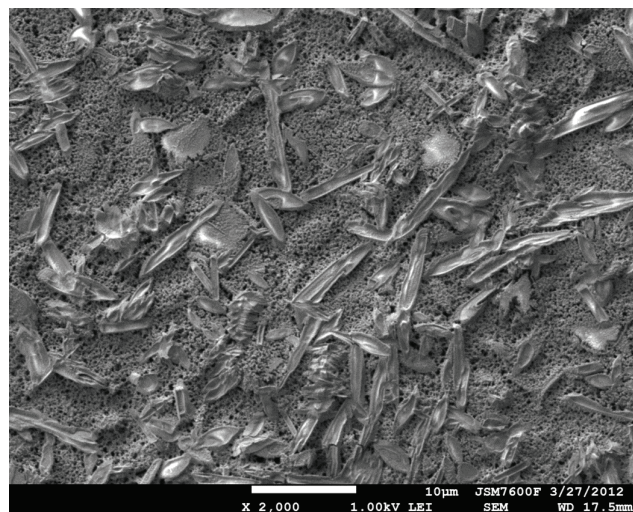
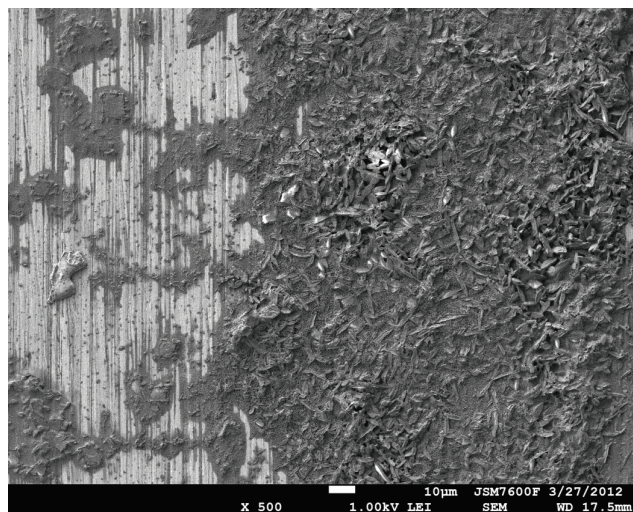


Figure 9. SEM images of DIT crystal with PEG1500: left: x500, right: x2,000.

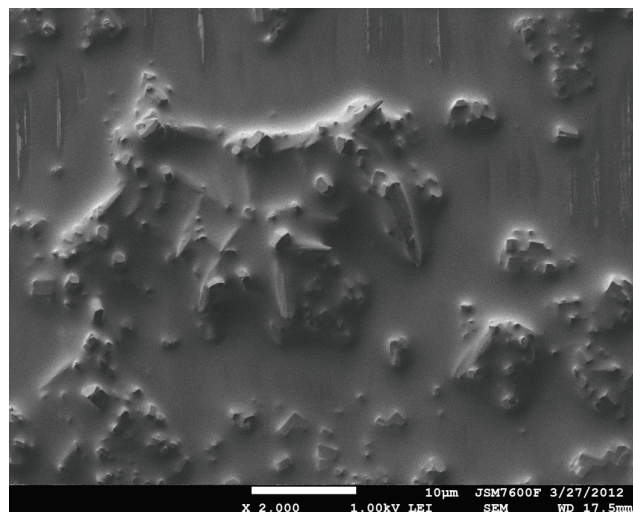
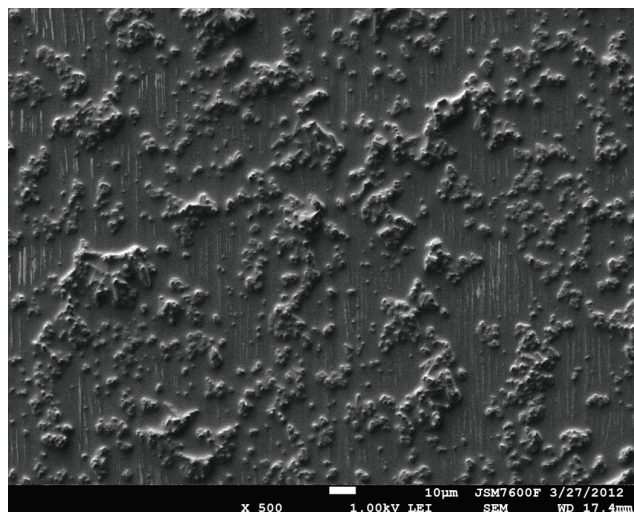


Figure 10. SEM images of IAA crystal with PEG1500: left: x500, right: x2,000.