

Application Note SECM370-3



Subject: Scanning Probe Application Sloping Area Scan Macro

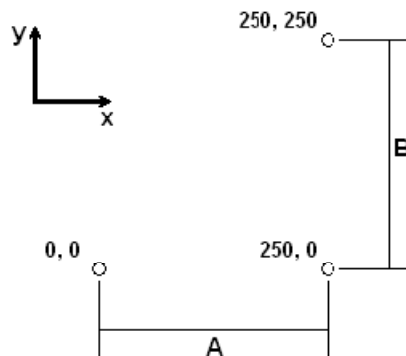
Scanning Electrochemical Microscopy Macro for Sloping Area Scan

Introduction

The SECM Sloping Area Scan macro allows a user to successfully account for slight variations in sample tilt. This is a macro-template combination which requires adding to the M370 root directory (i.e. c:\M370\). Setup, operation and common issues are discussed within this document.

This macro is designed for use in exactly the same context as the standard SECM area scan macro, with added functionality to define a sample's sloping nature. This can become necessary when the tip-sample distance is significantly smaller than the area over which you intend to scan. It is possible, with practice, to level a sample with the standard spirit-level to around 3 arc-minutes. This can be verified with the aid of the Tri-cell and the standard spirit level; the centers of the leveling screws are separated 250mm (on the short arms) and the sample can be judged to be level over a quarter turn of a leveling screw (which equates to 0.25mm vertical travel). In most cases, a 3 arc-minute slope would not contribute significantly to errors; however, in order to avoid sample contamination and/or sample damage it may not be possible to place a leveling device directly on top of the sample. Indirect leveling from other parallel-planar surfaces may then be required.

Performing three approach curves separated by a defined amount allows a user to calculate the expected vertical position of an exact current (here 10nA), and consequently, the angles between those points.



	A	B	C	D	E	F	G	H
1	x,y=	0,0			250,0			250,250
2								
3	micrometers	nanoamps		micrometers	nanoamps		micrometers	nanoamps
4	-80	14.9032		-79.4	14.897		-80.1	14.8469
5	-89.9	14.8657		-89.6	14.8594		-90	14.8031
6	-99.8	14.8156		-99.8	14.8219		-99.9	14.7594
7	-110	14.7594		-109.7	14.7531		-109.8	14.6906
8	-119.9	14.7031		-119.6	14.6718		-119.9	14.5717
9	-130.1	14.603		-129.8	14.5655		-129.8	14.4466
10	-140	14.4341		-140	14.4029		-139.7	14.2715
11	-150.2	14.2277		-149.9	14.1152		-149.9	14.0026
12	-160.1	13.8713		-159.9	13.6711		-159.8	13.5273
13	-170.1	13.2646		-169.8	12.9456		-169.7	12.6955
14	-180.1	12.2514		-180	11.7323		-179.9	11.2382
15	-190.4	10.0812		-185	10.3314		-185.2	9.60589
16	-195.6	8.03608		-189.9	8.58645			
17								
18		10nA @						
19	di	2.04512			1.74495			9.60589
20	dz	5.2			4.9			-185.2
21	di/dz	0.39329231			0.35611224			-0.0518677
22	D=(di/dz)Dz				D=(di/dz)Dz			D=(di/dz)Dz
23	Dz @10nA	0.20646221			0.93060546			7.59837683
24	Z=	-190.60646			-185.93061			-192.79838
25								
26			theta +x =	1.0715025		theta +y =	-1.5735815	
27								

FIGURE 1: The top diagram is a view from above, where the probe performs an approach at co-ordinate (0,0), then moves along 'A' and repeats at co-ordinate (250,0), and then again at (250, 250). The lower portion of this figure shows the three approach-curve data values and the calculation of the 'z' axis position of a 10nA current. Finally, the slopes in the '+x' and '+y' directions are calculated.

As an alternative to attempting to directly eliminate small samples tilts, this macro will scan across a defined and angled plane. This will require the user to perform three approach curves to similarly conducting or insulating regions of the sample, such that the sample slope can be extracted. Figure 1 above shows an example arrangement from which this information can be obtained.

The procedure to calculate the slope, as shown in Figure 1, is to choose a defined current within the approach curve data; here 10nA was chosen. Then calculate the slope between the bordering points (lines '15' & '16' for rows 'A' and 'B'): ' di/dz ' here was shown to be +0.393nA/ μm for the first approach curve. Finally, calculate the 'z' axis location of the 10nA current. Repeat this for the two remaining approach curve locations and use trigonometry to determine the slope between points.

Configuration and Setup

It is important to start with as-close an approximation to a level sample as possible, determine the slight offsets later by the approach curve technique, and account for these with this macro. In the extreme, the inhibition of the tip current will be reduced or negated as mediator will flow towards the tip from any accessible source. Similarly, effects from enhanced feedback or generated product from the sample will be less detectable on highly sloped samples.

After setting up the sample, electrodes and mediator, perform the approach curve experiments and enter the details in the configuration setup, as shown in Figure 2. The probe potential and experiment pre-delay allow for the probe to stabilize after turning on the electrochemical cell. The 'Line Delay' provides a method to minimize the effect of stirring after the probe returns from the end of a line.

The 'Sample Time' and 'Sample delay' provide a similar function, in that the sample delay is the time after the tip movement before the sampling starts, and the sample time is the period over which samples are acquired and averaged. The sample rate is set to 200Hz for the duration of this experiment – this provides a method by which the user can increase the signal to noise ratio to help detect low level signals.

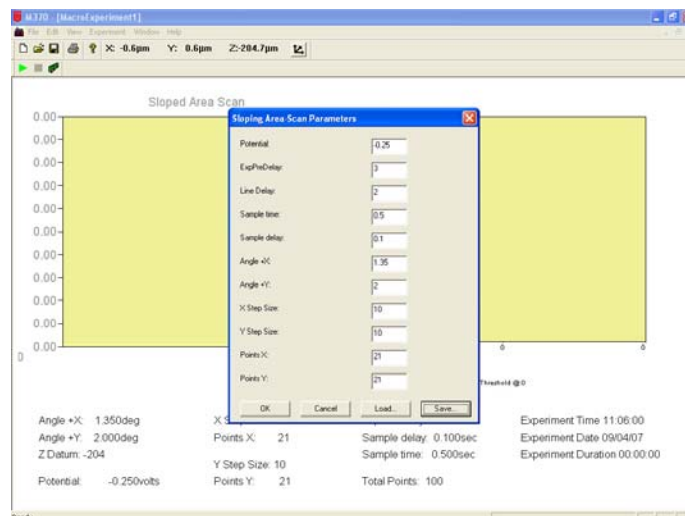


FIGURE 2: Configuration screen for Sloping Area Scan Experiment (V2.1.)

The 'Angle +X' defines the slope of the sample when travelling in a +x direction. In the case shown above, the slope is set to +1.35°, meaning that as the probe traverses towards +x, the sample is coming closer and closer to the probe; therefore, the probe should also move in a +z direction at the same time.

Finally, the 'x' and 'y' step sizes and total points define the area over which the probe will scan. On 'OK' of the configuration, it is possible to use the Experiment->Analysis menu function to view the expected course of the probe as it scans over the sample. Here, the z-axis location is plotted versus 'x' and 'y' co-ordinates. Figure 3 demonstrates this. In this figure, the z-datum is set to '-205 μm ', which reflects the position of the probe at the start of the experiment, and also, the bottom-left hand corner of the 'Analysis' function.

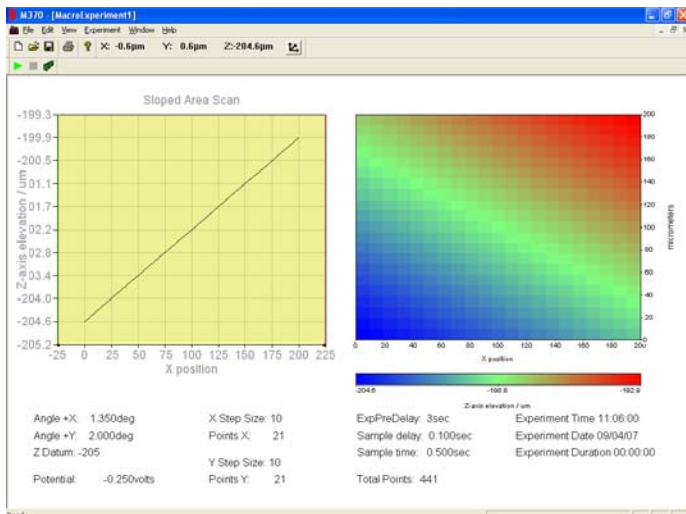


FIGURE 3: Experiment->Analysis function to show probe z-axis position during the course of a Sloping Area Scan experiment.

Finally, start the experiment and the probe will follow the given setup. Figure 4 shows a scan of a highly sloping sample (figure plotted in IsoPlot). Compare this to the picture shown at the start of this note, set on the same scales, which shows the macro's ability to handle topographic information in sync with the SECM area scan experiment.

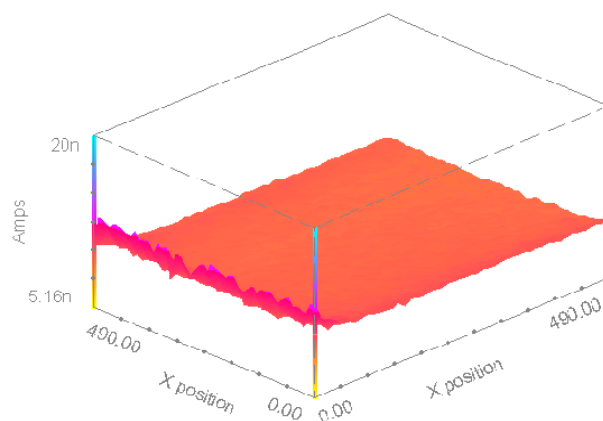
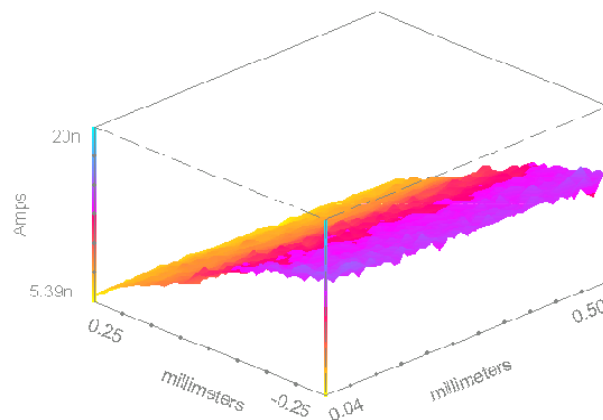


FIGURE 4: A.) An artificially highly-sloping sample set for demonstration purposes. B.) SECM experiments using this Sloping Area Scan Macro.