

# Coating/Sputtering Differences on SEM Stubs: User Guide

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## Introduction

Sputter coating in scanning electron microscopy (SEM) is the process of covering a specimen with a thin layer of conducting material; either a metal, such as a gold/palladium (Au/Pd) alloy, or carbon (C). A conductive coating is needed to prevent charging the specimen with an electron beam in the SEM high-voltage mode.

While metal coatings are also useful for increasing signal-to-noise ratio, they are of inferior quality when energy-dispersive X-ray spectroscopy (EDS) is employed. For this reason, when using EDS a carbon coating is preferred.

With the Hitachi TM3000 SEM, sputter coating is not required for all samples. Images up to a magnification of  $\times 4000$  are of high quality without any sputter coating when operating in the **Charge-up Reduction Mode (Settings > Observation Mode)**.

For detailed observations in magnifications greater than  $\times 4000$  and very delicate fossils, sputter coating before using the SEM can be used to potentially obtain higher quality images.

## Apparatus and Material

The sputter coater on the *JOIDES Resolution* is a **Leica EM ACE200** that supports carbon coating and Au/Pd sputtering. Other metal targets are supported as well; however, we are using only the Au/Pd system at this time.

The following quick introduction should give an overview of the different coating options and the resulting layer of conductive material. The sample material for this test is sand. The following coating options were selected as shown in Tables 1 and 2.

Material: Gold-Palladium					
No.	Rotation speed	Coating direction	Coating time (sec)	Purge cycles	Coating time (min)
1	1	Directional	15	2	7
2	1	Directional	25	2	10
3	1	Diffuse	30	2	7
4	1	Directional	25	5	9
5	1	Diffuse	30	5	7

**Table 1. Au/Pd coating settings.**

<b>Material: Carbon</b>			
No.	Threading	Pulse/flash	Coating time (min)
6	Single	1 pulse	3
7	Single	5 pulses	3
8	Single	2 flashes	3

**Table 2. Carbon coating settings.**

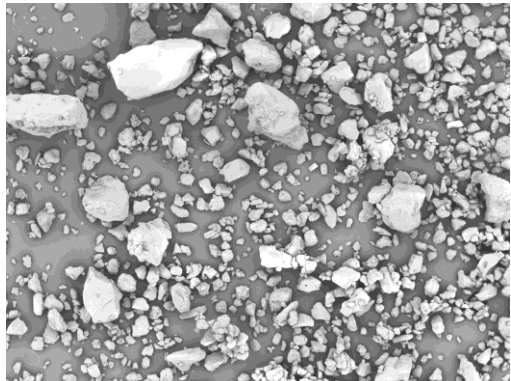
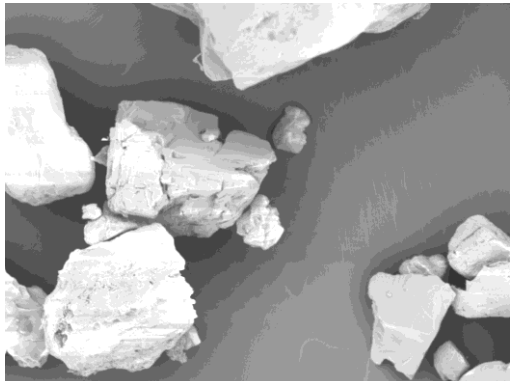
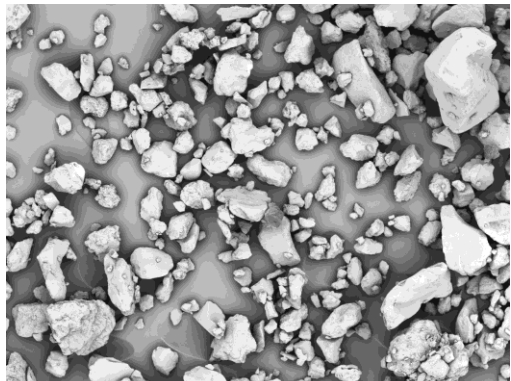
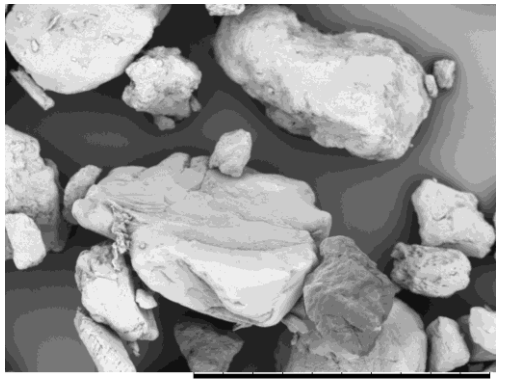
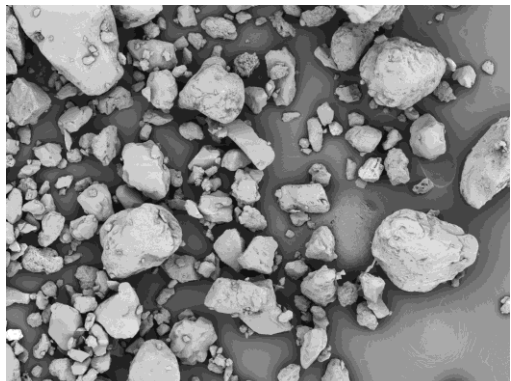
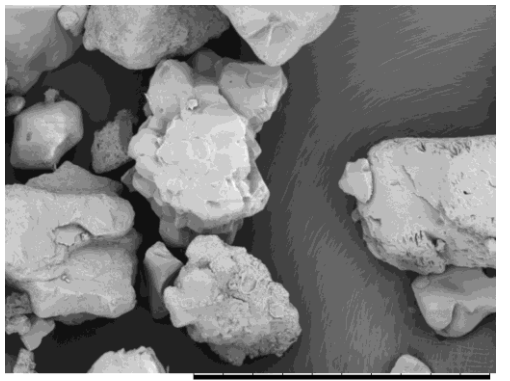
The venting process after each sputter coating process takes 99 sec.

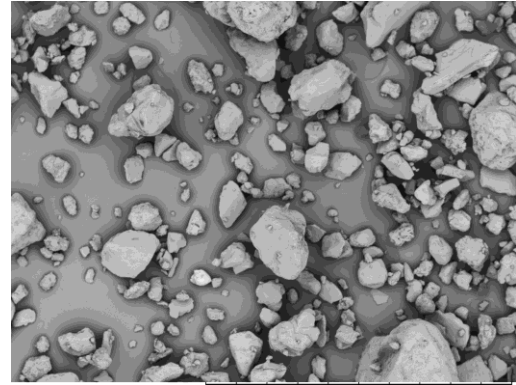
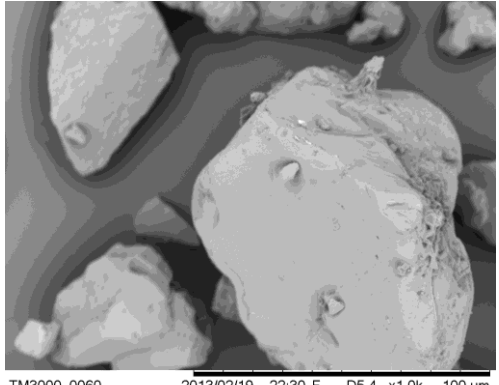
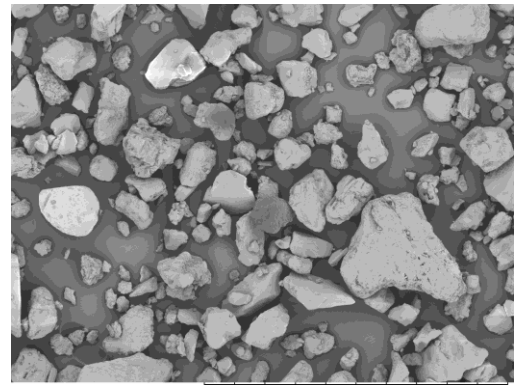
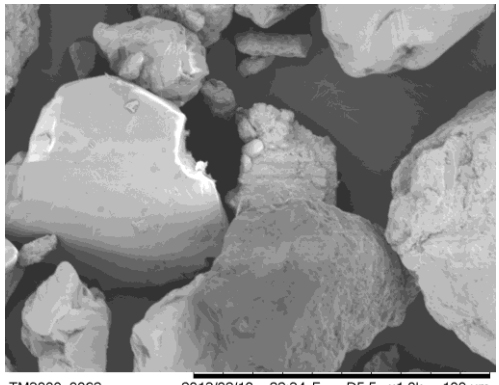
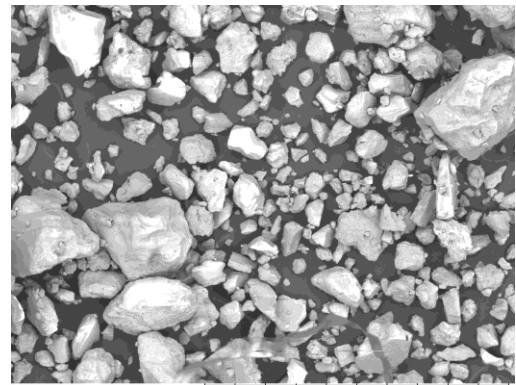
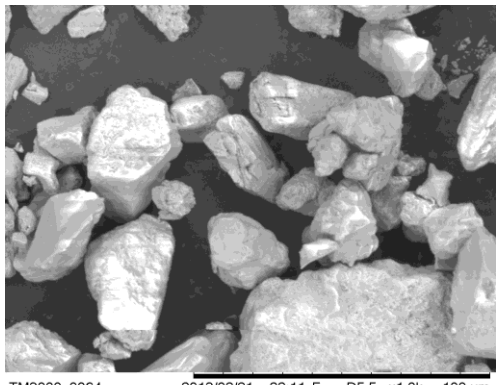
Figure 1 shows the coated stubs. The stub in the upper left corner was not sputtered, numbers 1–8 were sputtered with gold-palladium and/or carbon. The sputtering parameters are listed above in Tables 1 and 2.



**Figure 1. Coated Stubs.**

Each stub was checked for image quality using the Hitachi SEM TM3000, as shown in Figure 2. Note that Figure 2 is a multi-page figure.

<p>No. 1, x200</p>  <p>TM3000_0053 2013/02/19 22:10 F D5.3 x100 1 mm</p>	<p>No. 1, x1000</p>  <p>TM3000_0054 2013/02/19 22:12 F D5.3 x1.0k 100 um</p>
<p>No. 2, x200</p>  <p>TM3000_0055 2013/02/19 22:14 F D5.3 x200 500 um</p>	<p>No. 2, x1000</p>  <p>TM3000_0056 2013/02/19 22:15 F D5.3 x1.0k 100 um</p>
<p>No. 3, x200</p>  <p>TM3000_0057 2013/02/19 22:19 F D5.3 x200 500 um</p>	<p>No. 3, x1000</p>  <p>TM3000_0058 2013/02/19 22:20 F D5.3 x1.0k 100 um</p>
<p>No. 4, x200</p>	<p>No. 4, x1000</p>

 <p>TM3000_0059 2013/02/19 22:28 F D5.4 x200 500 um</p>	 <p>TM3000_0060 2013/02/19 22:30 F D5.4 x1.0k 100 um</p>
No. 5, x200	No. 5, x1000
 <p>TM3000_0061 2013/02/19 22:33 F D5.5 x200 500 um</p>	 <p>TM3000_0062 2013/02/19 22:34 F D5.5 x1.0k 100 um</p>
No. 6, x200	No. 6, x1000
 <p>TM3000_0063 2013/02/21 22:09 F D5.5 x200 500 um</p>	 <p>TM3000_0064 2013/02/21 22:11 F D5.5 x1.0k 100 um</p>
No. 7, x200	No. 7, x1000



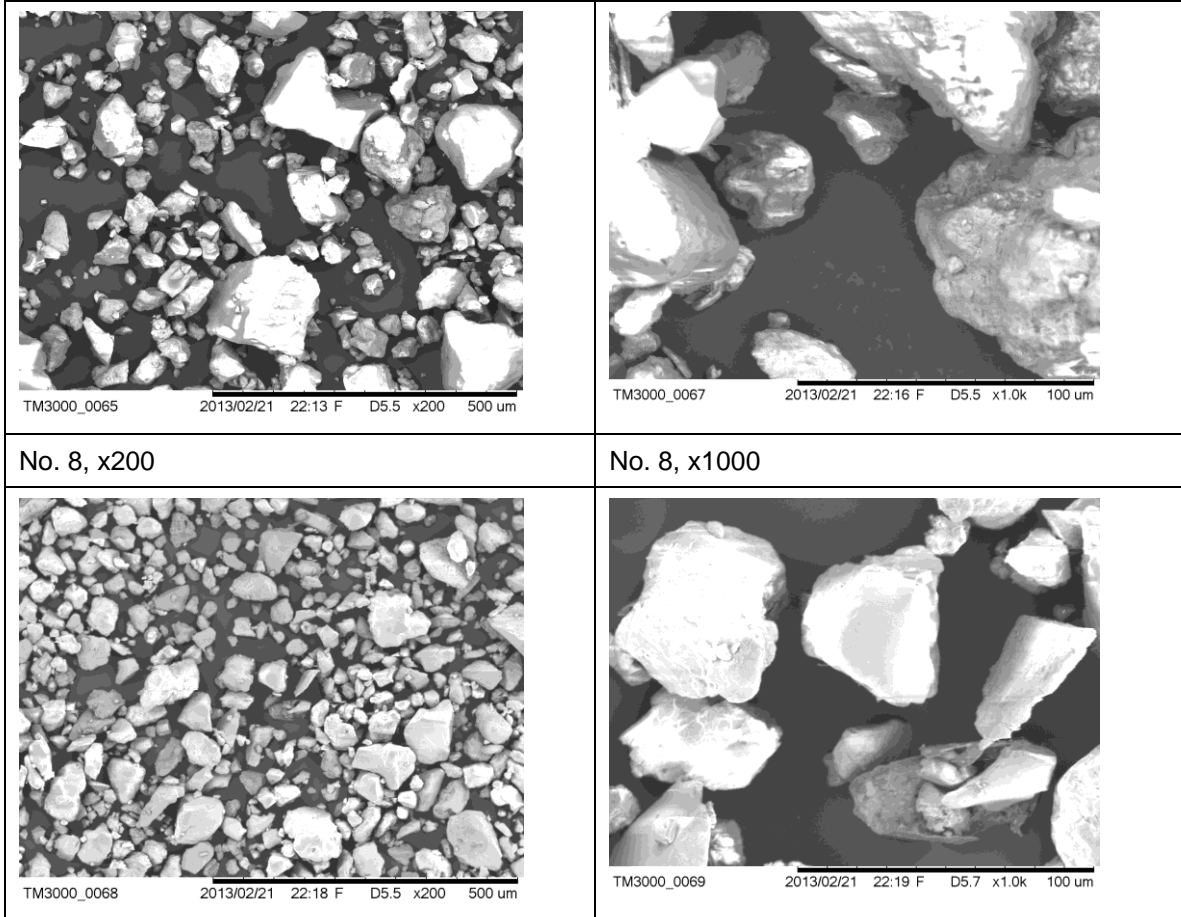


Figure 2. SEM Images of Coatings.

## Observations

- Sputter coating with gold-palladium results in a more evenly distributed thin layer compared to carbon thread sputtering.
- When sputtering with metal, purge cycles of >2 provide a better distribution of the metal.
- A sputter duration between 25 and 30 sec gives the best results in metal distribution and thus provides the best images.