Comparison of Copper Diallyl Phthalate and Conductotherm 3000 Sample Mounting Media

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Instrument: GDS500A

Background

Samples less than the diameter of the lamp o-ring require mounting in an electrically conductive media. Glow discharge spectrometry with a direct-current lamp requires a vacuum-tight seal and a conductive sample.

- The standard 4 mm lamp requires a sample diameter of at least 15 mm or larger; smaller samples require mounting. An optional 2 mm anode can also be used with this lamp, but still requires a mounted diameter of 15 mm.
- The optional small 2 mm lamp requires a sample diameter of 9 mm or larger; smaller samples require mounting.
- Performance Note 209-076-031 discusses many different sample sizes and how to prepare each one for analysis.

Making a mount requires the sample to be placed in a metallographic mounting press with the analytical surface facing down. Mounting media is added on top. After the heating/pressure cycle, the sample is removed encased in a solid mount. For bulk analysis samples, the surface to be analyzed is ground using a belt grinder or wet wheel polisher. If the sample is to be analyzed with compositional depth profiling, no further sample preparation should be performed. The mounted sample is now ready to be placed on the glow discharge lamp. Position the specimen directly over the anode opening where it can be excited. Examine the sputter crater to ensure the entire analysis took place on the sample and not the media.

Media Recommendations

Cu-diallyl phthalate mounts work on each lamp configuration. The copper is dense and helps dissipate the heat produced during the analysis. It is expensive in relation to other metallographic mounting materials because it contains copper. A cooling puck or block can be used on the back. If the media is sputtered, then a high copper concentration might be seen, but a short circuit will usually occur before the analysis is complete. The press will require cleaning, as this copper-based media sticks to the platen and ram.

Conductotherm 3000 is a carbon-based mounting material that has been used less in the past. The graphitic material is electrically conductive, but is not as good at keeping the sample cool as the copper diallyl phthalate. The cost is less than the copper-based media and the mounts are of high quality. The small 2 mm lamp can be used for analysis of samples mounted in this material. Problems were noticed using the 4 mm lamp; depending on sample size and mass, the mount can crack, which causes a vacuum leak. An overheated sample gives erroneous results such as the carbon being high. When using the 4 mm lamp equipped with a 2 mm anode, arcing to the edge of the anode causes shorts. If using the 4 mm lamp, the sample should cover the blue o-ring completely and be of sufficient mass to not overheat. This media can be used with normal cleaning of the press parts.

Recommended Weights

Copper diallyl phthalate

- 1 lb container (PN 811-138) and 5 lb container (PN 811-139)
- Amount: 50 g for a 1.25 in mount and 80 g for a 1.5 in mount (the provided scoop holds approximately 40 g or 15 mL)
- Follow the heating and pressure cycle times on the container

Conductotherm 3000

- 2.2 lb (1000 g) container (PN 812-233)
- Amount: 20 g for a 1.25 in mount and 30 g for a 1.5 in mount (the provided scoop holds approximately 40 g or 45 mL)
- Follow the heating and pressure cycle times on the container

Spectroscopy Performance Note

Spectroscopy Performance Note

Mounting Tips

- Taller materials require additional mounting material to ensure complete encapsulation of the part.
- Grind a flat surface before mounting if possible.
- Center the sample on the platen, add loose media on all sides evenly.
- Be aware that the mounting media contains loose powder and dust. Please take safety precautions to safe guard against exposure to dust particulates while mounting and grinding.
- It is the responsibility of the user to take the proper safety and health practices and determine appropriate action before attempting this procedure.

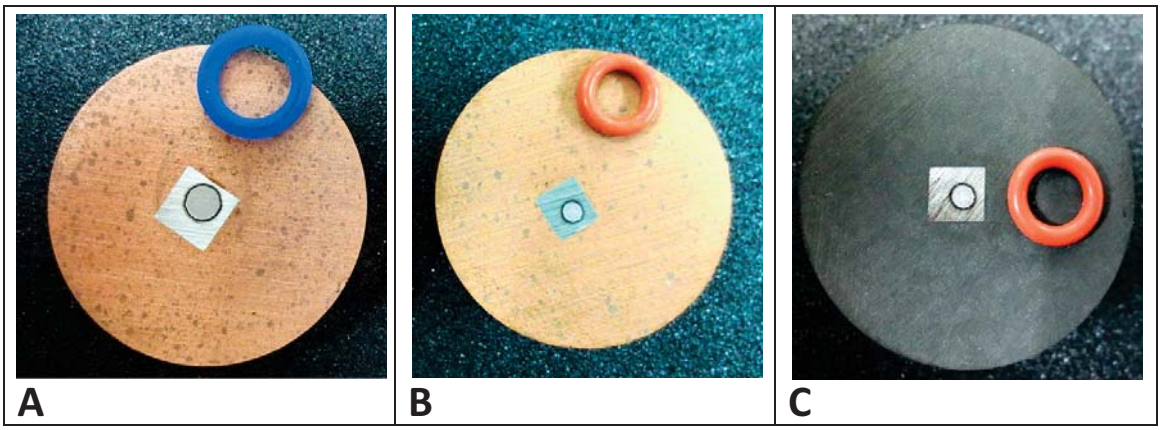

FIGURE 1: Mounted samples (A) Copper Diallyl Phthalate with 4 mm anode, (B) Copper Diallyl Phthalate with small 2 mm lamp/anode, and (C) Conductotherm 3000 with small 2 mm lamp/anode.

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