Dimpling ductile materials

Technical note

Why dimple TEM specimens?

Dimpling has become the preferred first step in transmission electron microscope (TEM) specimen preparation because (1) it greatly reduces the time necessary for final thinning to electron transparency (the time saving is especially great when the final polishing step is relatively slow as is the case in ion polishing); (2) the electron transparent area will always form at the center of the specimen disc and hence less x-ray shadowing will occur during energy dispersive x-ray spectroscopy (EDS) analysis and less specimen movement will occur during tilting; (3) the thick, supportive rim of material formed during dimpling greatly facilitates the handling of fragile, TEM specimens.

Dimpling ductile materials

We are frequently asked by our customers to recommend a procedure for dimpling ductile materials, such as pure metals and solders. The problem usually encountered is that the cutting rate is low in comparison to that achieved with harder materials such as metal oxides and semiconductors. The reason for this is that the polishing grit (usually diamond or aluminum oxide) embeds itself in the soft material during dimpling and then the dimpling wheel starts to be ground away rather than the specimen. The solution to the problem is to use cubic boron nitride (CBN) as the polishing compound. CBN particles have a rounded shape and do not become so easily embedded in the materials being polished.

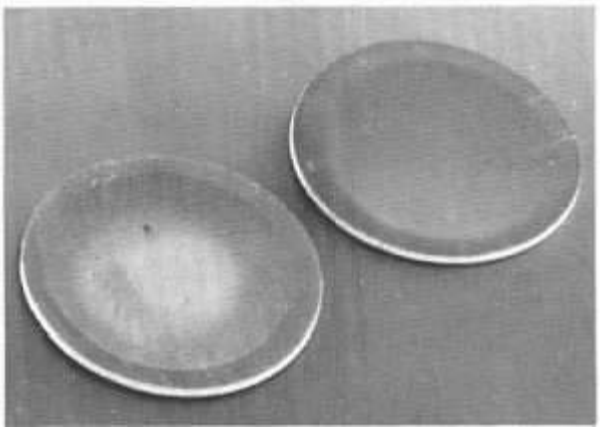

Figure 1. SEM image of a 316 stainless steel TEM specimen disc after grinding (left) and polishing (right) with CBN.

Recommended procedure

We recommend that the starting thickness of the ductile material be somewhere in the range 100 – 150 µm. Cut 3 mm diameter specimen discs from this material using the model 659 Disc Punch from Gatan. Fit the model 656 Dimple Grinder with a 15 mm or 20 mm diameter phosphor bronze wheel, then dimple the 3 mm discs using 4 – 6 µm CBN grit down to a thickness of about 10 µm. Install a felt wheel and continue polishing first with 4 – 6 µm, and then with 0 – 2 µm CBN grit until the coarse marks formed during grinding by the 4 – 6 µm grit have all been removed. Use sperm whale oil substitute as a lubricant to keep the CBN paste from drying out during both the grinding and polishing steps.

Measurement of cutting rates

Aluminum, stainless steel, copper and a Pb-Sn eutectic solder were each thinned from about 100 to <10 µm with 4 – 6 µm CBN paste, a dimpling load of 10 g, a wheel speed of 150 rpm, and a wheel diameter of 15 mm. The bar graph shows the various cutting rates obtained. It can be seen that CBN cuts soft materials at about 2 – 6 µm/min, which is the same rate that diamond cuts silicon under similar conditions.

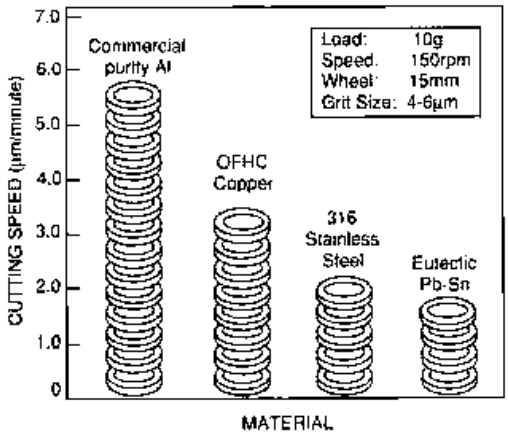

Figure 2. Dimpling rates for some common ductile materials using CBN paste.

Ordering

Gatan provides CBN paste in convenient 5 g syringes.

0 – 2 µm CBN: 656-04-010

4 – 6 µm CBN: 656-04-01 1