Twin-jet electropolishing of Ni-Ti microwires

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Twin-jet electropolishing is a fast, cheap and clean method to prepare TEM specimens of alloys. However, the conventional twin-jet electropolish setup is designed for discs of 3mm diameter material which makes it hard to be used for electropolishing materials of smaller size.

The aim of this work is the preparation of TEM specimens of 150m diameter Ni-Ti wires with nano-size grains. Cross-sectional Focused Ion Beam (FIB) sample preparation could be used for this purpose, but apart from the small size of FIB samples which does not allow proper statistical investigation of, e.g., grain size and orientation, FIB induced damage hampers a proper interpretation of the images.

To perform electropolishing on a 150m diameter microwire, a piece of about 2mm length of wire was cut and embedded in a drop of EPO-TEK 353ND resin, making a hemisphere with a diameter of about 3mm. In order to make the resin non-transparent so that the conventional perforation detection can be used, some graphite powder is added to the resin. After the resin is cured, it is mechanically grinded from both sides until a thin disc of resin containing the microwire is obtained. For a 150m wire to appear wide enough from both sides of the resin disc, it has to be thinned down to about 80m, as shown in Figure 1 (a). The resin disc embedding the wire is then gently mounted into the twin-jet electropolisher holder in the way that the wire connects to the electrical contact of the holder (although the resin contains graphite its electrical conductivity was found to be too low). The holder is then inserted into the electropolisher and the electropolishing procedure starts. In the present case a mixture of 80% methanol and 20% sulfuric acid operating at 130 mA and 0°C in a Struers Tenupol twin-jet electropolishing equipment was used. As the resin-wire complex is very thin and fragile and the wire has a tendency to detach from the resin, the minimum flow rate which the electrolyte still reaching the sample (4.5) is set. Moreover, as the wire is quite thin, the maximum possible photosensitivity of the instrument (8) is set to react immediately after perforation to prevent the wire from dissolving completely. The opaque resin blocks the light-photodetector path and the wire is electropolished until perforation occurs, as can be seen in Figure 1 (b). The latter implies that the central parts of the wire have been completely removed but TEM images clearly show that the remaining opposite sides of the microwire show the needed electron transparency. Finally, to prevent charge build-up on the non-conductive resin during TEM observation, a layer of 5nm carbon is deposited on both surfaces of the resin. The obtained specimen is appropriate for electron microscopy, of which two examples showing different nanograin sizes are presented in Figure 2.

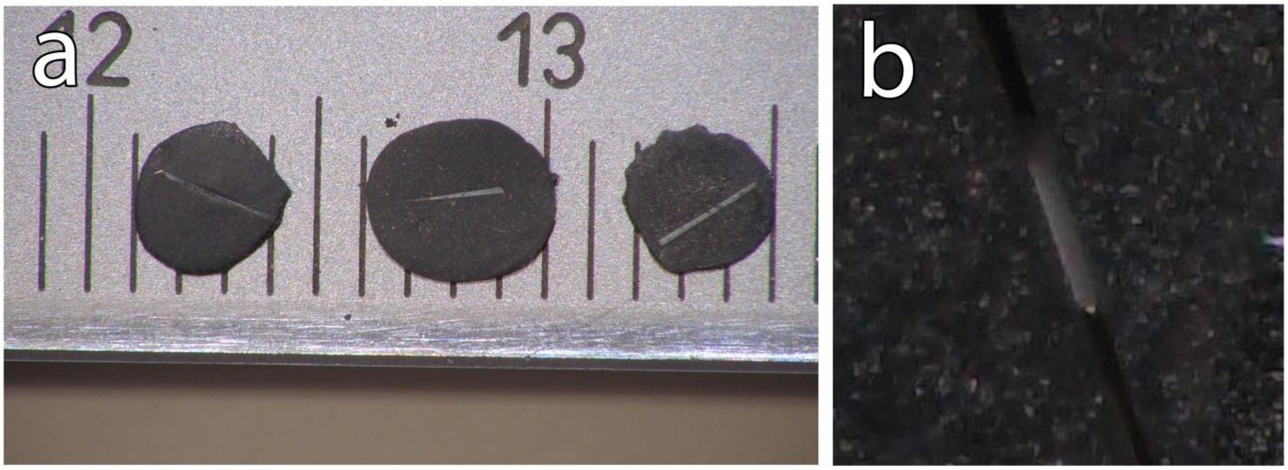

Figure 1 (a) Ni-Ti microwire embedded in resin and grinded to 80μm thickness; (b) Electropolished microwire with hole in the center of the microwire.

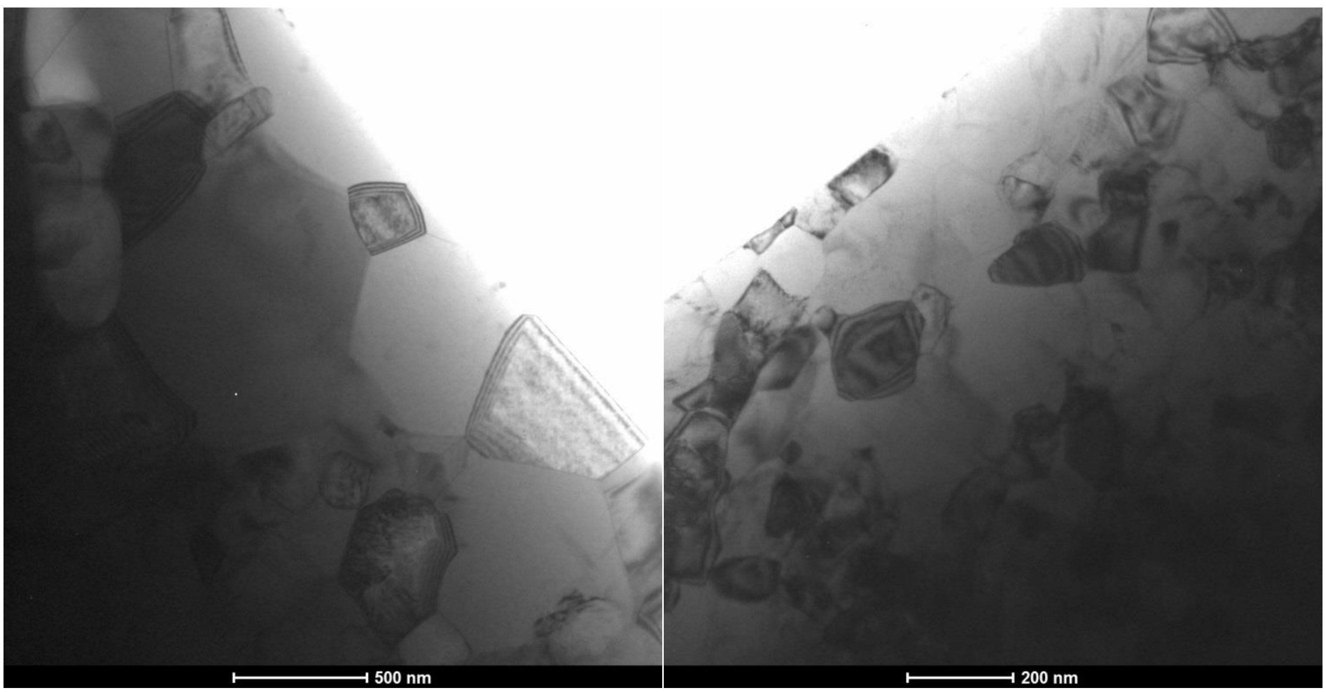

Figure 2 Two Bright Filed TEM images from microwire specimens prepared by the introduced method.

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