

Electro-Polishing Foil Samples for TEM with an Extremely Small Amount of Electrolyte

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An innovative method to electro-polish metallic material to electron transparency, where only an extremely small amount of electrolyte (less than a few ml) is used, is developed and applied to a few metallic materials.

The principle of the method is illustrated in Fig.1. A conventional 3mm ϕ disc is sandwiched between a pair of ring foils (**1** and **2**; 0.15mm thick, stainless steel), together with a spacer (**3**; 0.15mm thick, stainless steel). This assembly is mounted into an electro-polishing cell (Fig.2), which is in turn mounted to the electro-polisher.

The electro-polishing cell is connected to the anode via a guide **G** and a needle cathode is lowered, the distance between the tip of the cathode and the sample being adjusted by observing under optical microscope [1]. Electrolyte is poured with a pipette and electro-polishing starts.

The lower surface of the sample is monitored by an optical microscope [2]. The first step is to electro-polish the upper surface of the sample, which is interrupted well before a hole is penetrated. Then the cell is turned over and the other (bottom) surface is polished while the now polished upper surface is being observed by an optical microscope [3] and displayed on a TV screen (Fig.3). The polishing is finalized when a small hole is perforated in the center: The sample is dismantled and cleaned in a usual manner.

Fig.4a shows a DF image of 17-7PH steel taken with a forbidden spot shown in the diffraction pattern. Fig.4b shows a HREM of the precipitate in Fig.4. The precipitate shows lattice twice as large as the matrix. Fig.5 shows EDX analysis; the precipitates are identified as β' -NiAl in good agreement with [1]. Fig.6 shows precipitate of Ni₃Al in a Ni-base super-alloy. These micrographs were taken in a Cs corrected microscope ARM operated at 200kV.

References:

[1] N Yukawa, M Mizutani and H Saka, 6th International Congress for Electron Microscopy, Kyoto(1966) p.403.

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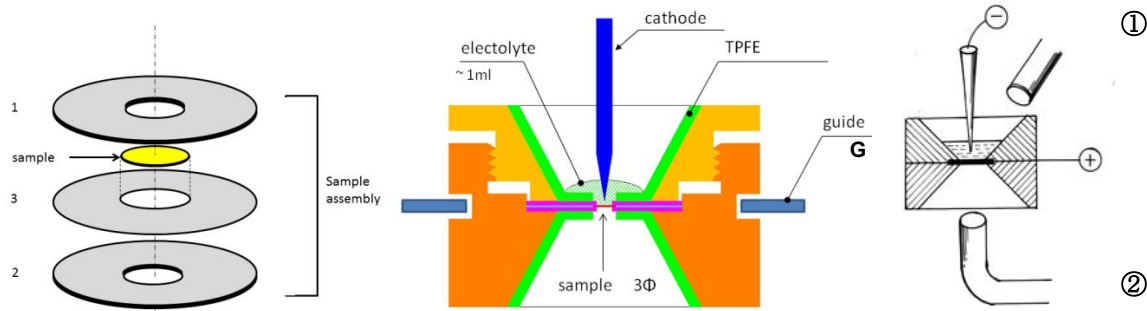


Figure 1. Sample assembly. **Figure 2.** Electro-polishing cell. **Figure 3.** Monitoring

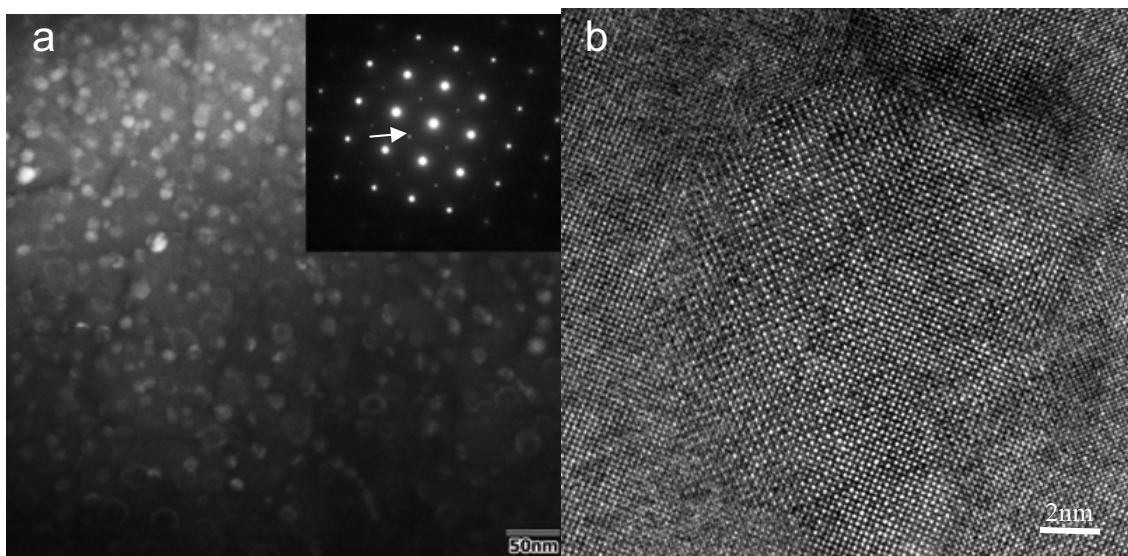


Figure 4. (a) DF image of 17-7PH stainless steel taken with a forbidden spot (indicated by arrow) in the diffraction pattern (inset). (b) HREM of a precipitate.

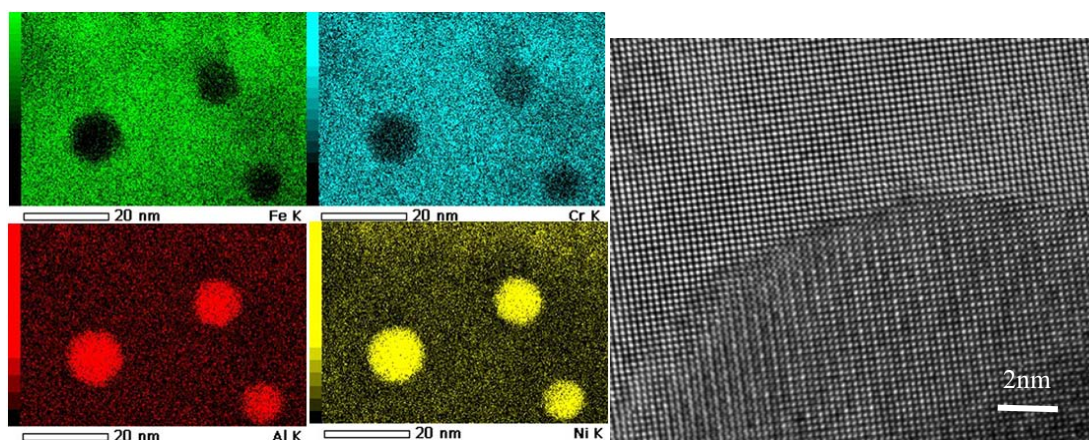


Figure 5. EDX mapping

Figure 6. HREM of γ' in Ni-Al.