doi:10.1017/S1431927613010933
Microsc. Microanal. 19 (Suppl 2), 2013

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

H.Saka[1], M.Yamamoto[2],Y.Shiraishi[2], M.Amano[2], T.Goto[2],T.Suzuki[3], S.Arai[3] and K.Sasaki[1]

[1.]Nagoya University, School of Engineering, Nagoya, Japan
[2.]Yamamoto Chemical Co, Hiroshima, Japan
[3.]Nagoya University, Ecotopia Science Institute, Nagoya, Japan

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.

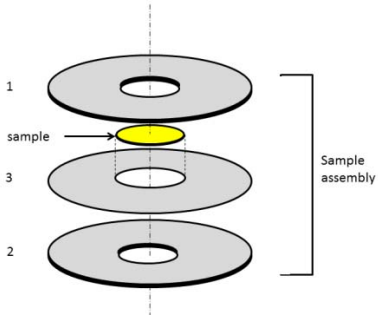
The principle of the method is illustrated in Fig.1. A conventional 3mm φ disc is sandwiched between a pair of ring foils (**1** and **2**; 0.15mm thick, stainless steel), together with a spacer (**3**;0.15mm thick, stainless steel). This assembly is mounted into an electro-polishing cell (Fig.2), which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

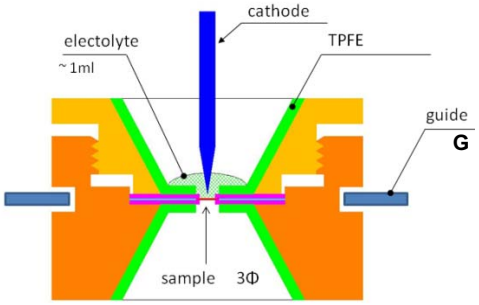
The electro-polishing cell is connected to the anode via a guide **G** and a needle cathode is lowered, the distance between the tip of the cathode and the sample being adjusted by observing under optical microscope **[1]**. Electrolyte is poured with a pipette and electro-polishing starts.

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 dismounted 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 Ni_3Al 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.
[2] This work was carried out in cooperation with Excellent Support Center for Reaction, Nanomaterials and Biological Science by Electron Microscopy, Nagoya University.
[3] H. S thanks the Ministry of Education, Culture, Sports, Science and Technology for the financial support (Grant-in-Aid for Scientific Research (C), Contract #24560803).





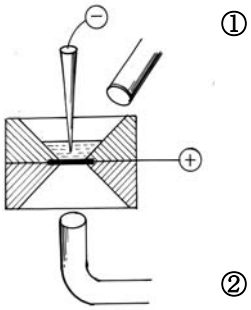


Figure 1. Sample assembly. **Figure 2.** Electro-polishing cell. **Figure 3.** Monitoring

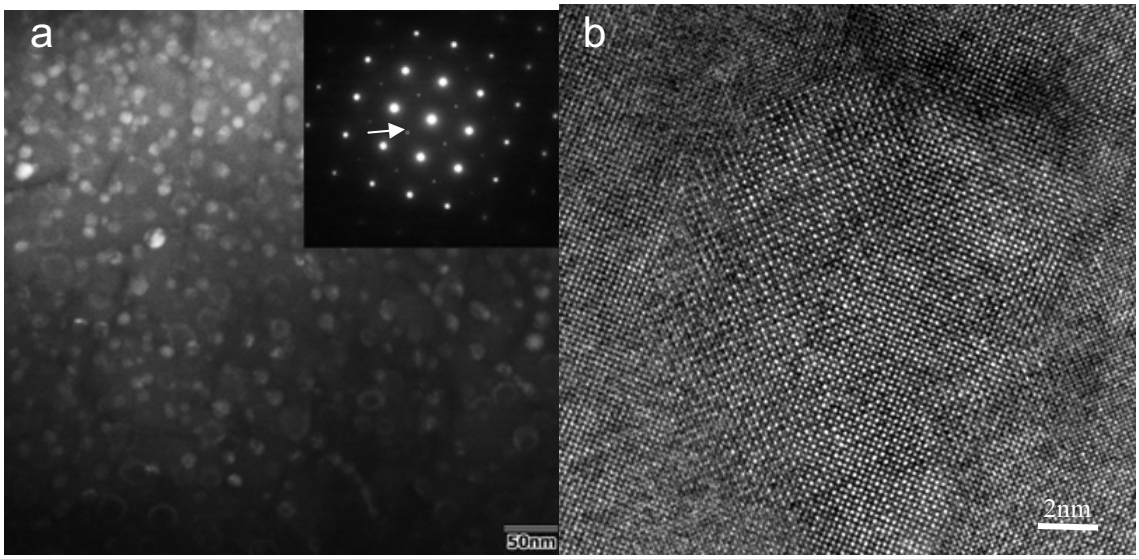
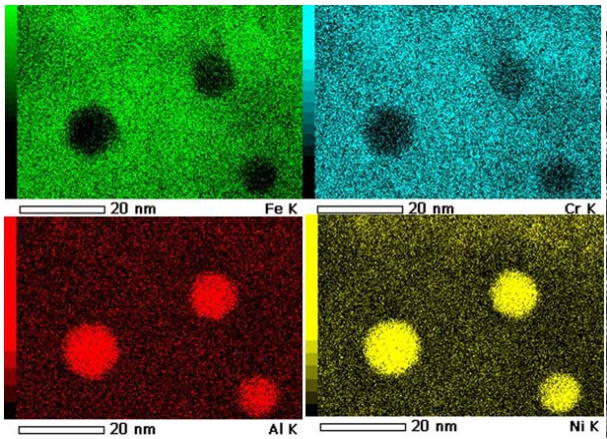


Figure 4. (a) DF image of 17-7PH stainless steel taken with a forbidden spot (indicated by arrow) in the diffraction pattern (inset). **(b)** HREM of a precipitate.



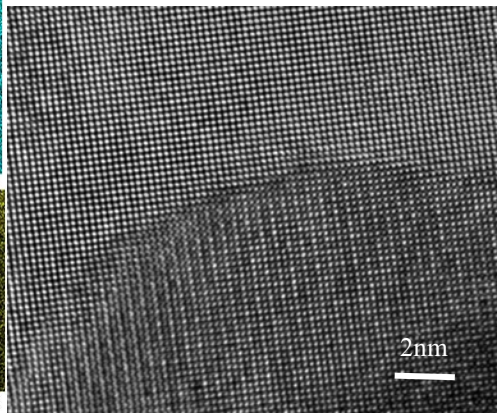


Figure 5. EDX mapping **Figure 6.** HREM of γ ' in Ni-Al.

https://doi.org/10.1017/S1431927613010933 Published online by Cambridge University Press