

# Identification of Secondary Phases in a Ti-Mo Alloy

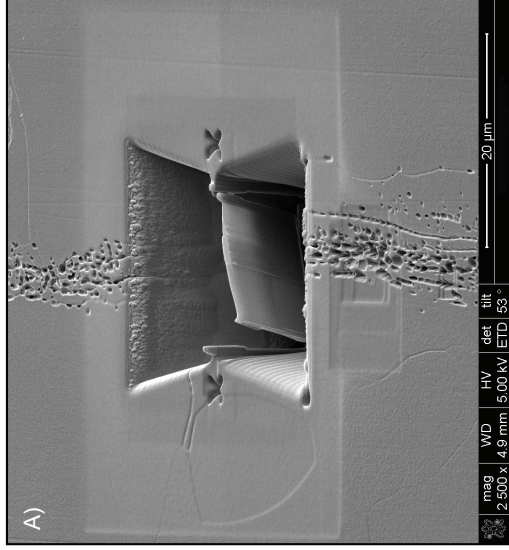
**Class 3:** Electron Microscopy – Transmission and Analytical

**Alloy:** Ti-Mo meta-stable beta titanium alloy

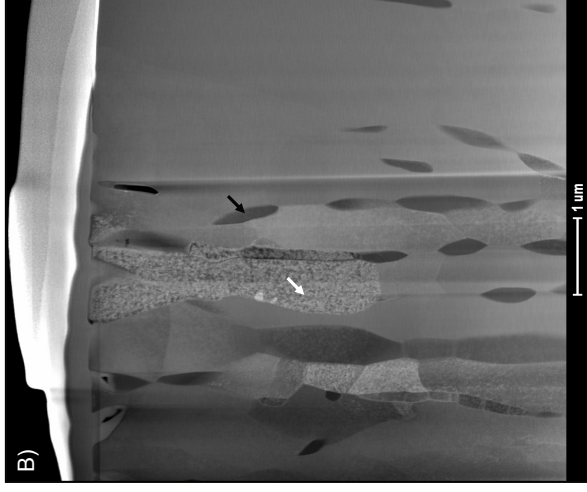
**Etchant:** n/a

**Equipment:** Nova 600 DB-FIB and Tecnai F20 S/TEM

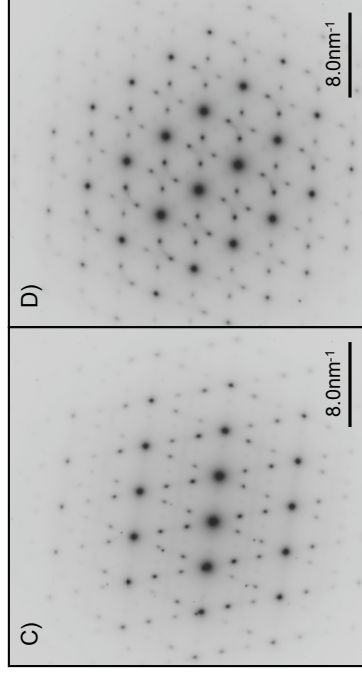
An undesirable band of precipitates were observed optically along the rolling direction of the sheet after final heat treatment. Since scanning electron microscopy was ineffective due to the size of the suspect features and because of the dissolution of features by the chemical etchant (i.e. pitting), S/TEM was selected for phase identification. To circumvent the etchant issue and to prepare a TEM specimen containing the suspect features, a FIB area using the etch pits as a guide. After thinning, the 10 x 10  $\mu\text{m}$  membrane was lifted out from a region that contained a significant fraction of the suspect features (see Figure A). Selected area electron diffraction patterns were obtained from matrix grains in Figure B (highlighted with white arrow) and the unknown precipitate features (high-lighted with black arrow). The large, matrix grains exhibited a BCC crystal structure and lattice parameter consistent with beta phase. The mottled appearance of these matrix grains was due to very fine omega precipitates, which were discovered incidentally by the extra-lattice reflections (Figures C&D). Finally, diffraction patterns of the unknown features (Figures E&F) confirmed that the dark precipitates observed during optical microscopy were alpha phase. The material was re-processed using a modified heat treatment to dissolve the undesirable precipitates without grain coarsening or degradation of mechanical properties.



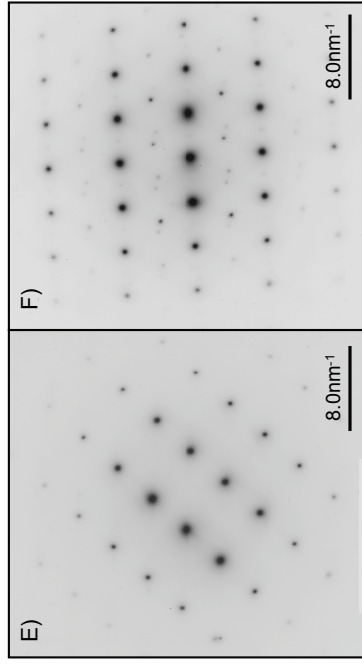
A) Secondary electron image from DB-FIB illustrating the nearly-detached TEM sample which contains a significant fraction of the suspect features. Rolling direction is vertical in image.



B) HAADF STEM darkfield image (200kV) of the 10 $\mu\text{m}$  x 10 $\mu\text{m}$  wafer extracted by FIB. Areas of interest are the mottled, large grains (white arrow) and the dark features (black arrow).



Selected Area Diffraction Pattern from the 'mottled' grain highlighted with white arrow in Figure B, which corresponds to the body-centered cubic beta phase.  
C) Zone Axis=[1 1 3]. The sharp extra reflections at 1/3 and 2/3 positions are due to the hexagonal omega phase.  
D) Zone Axis=[0 1 1]. The distinctive 'X' patterns are extra reflections due to the hexagonal omega phase.



Selected Area Diffraction Pattern from suspect feature highlighted with black arrow in Figure B, which was identified as alpha phase.  
E) Zone Axis=[0 1 -1 2]  
F) Zone Axis=[0 1 -1 0].