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**Enabling Science and Technology through
European Electron Microscopy**

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Deliverable 6.2

Report on protocols and standards developed in ESTEEM2

Deliverable leader 6 – Max Planck Institute for Intelligent Systems, Stuttgart

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**Report on protocol for TEM sample preparation of intermetallic
Ti-Al based alloys (3rd generation).**



TEM sample preparation of Ti-Al based alloys

Intermetallic Ti-Al based alloys are promising candidate for future high temperature structural material applications (i.e. turbine blades, turbocharger wheels). Main reason for this is their low density, high stiffness, high strength to weight ratio, good oxidation resistance at high temperatures, good creep properties.

The γ -TiAl alloys with Nb addition (third generation) were developed to increase the operational temperature of gas turbines by replacing conventional titanium-base alloys. They are additionally foreseen as an alternative for heavy nickel-base superalloys in moderate temperature range (to about 800 °C), because of high strength to weight ratio.

Steps of TEM specimen preparation of a Ni-base superalloys:

The steps of specimen preparation may be divided into four main parts:

- Initial thinning to the thickness of about 80 μ m ,
 - Cutting of a 3 mm disc,
 - Dimpling
 - Final thinning: electropolishing, followed by ion-beam milling (if necessary)
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- In the first step, slice(s) about 1 mm thick should be cut. During process of cutting, the specimen and cutting disc should be cooled i.e. with water.
 - The specimen (glued to the holder) should be grinded on sandpapers (starting from about 400, through 600, 800 and finishing on 1000 grid to about half of its thickness.
 - The specimen should be unglued, cleaned with acetone. The thickness should be measured and the second side of the specimen should be grinded, again, starting from 400 till the 1000 sandpaper grid. During the process, thickness should be controlled and the remaining thickness of the slice should be around 80 μ m.
 - After that, the specimen should be unglued, cleaned with acetone and ethanol.
 - 3mm disc should be cut from the cleaned specimen using a disc puncher.



- In the next step, the specimen should be dimpled to the remaining thickness of about 60 μm .
- The next step of TEM specimen preparation is electropolishing of a 3mm disc. The method is basing on anodic dissolution of the examined specimen. A twin jet apparatus, (e.g. Tenupol 5 of Struers) is used to pump electrolyte jet at both sides of a specimen disc. There is a light beam and a light sensor, which detects transparency (hole) of the specimen and shut off the process of electropolishing.

During electropolishing of TiAl based alloys (using Tenupol 5 of Struers), the following conditions may be used:

electrolyte: A3 of Struers

temperature of the electrolyte: +5 °C

flow rate: 10 (arbitrary unit – different in different polishing units)

voltage: 20 V

- If necessary, additional ion milling (e.g. using PIPS of Gatan) may be performed. The following conditions may be used: at 3.5keV, 4deg, time of the thinning should be adjusted for the specimen thickness.

Specimen (thin foil) is ready for TEM investigations.