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**Deliverable 6.2**

**Report on protocols and standards developed in ESTEEM2**

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**input to Deliverable 6.2:**

**Report on protocol for TEM sample preparation of CM186LC,  
a second generation DS nickel based superalloy**



## TEM sample preparation of CM186LC superalloy

Nickel based superalloys exhibit very high strength at high temperatures, creep- and oxidation resistance. They are widely used in extreme environments, mainly in aeronautics, aerospace (e.g. parts of jet engine's turbine blades) and power generation (e.g. stationary gas turbines) industry.

CM186LC is a second generation Directionally Solidified (DS) superalloy. Its nominal composition is the following (wt %):

C	Cr	Co	Mo	W	Ta	Re	Al	Ti	B	Zr	Hf	Ni
0.07	6	9	0.5	8	3	3	5.7	0.7	0.015	0.005	1.4	Bal.

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

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

- Initial thinning to the thickness of about 100µm,
  - Cutting of a 3 mm disc,
  - Final thinning: electropolishing,
  - Ion-beam milling.
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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, 1000 and finishing on 1200 grit to a thickness of few hundred µm.
  - 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 sandpaper 1200 grid. During the process, thickness should be controlled and the remaining thickness of the slice should be lower than 100µm.
  - After that, the specimen should be unglued, cleaned with acetone and ethanol.



- 3mm disc should be cut-off using a disc puncher.
- The next step of specimen preparation is electropolishing of a 3mm disc. The method is based 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 a CM186LC superalloy (using Tenupol 5), the following conditions may be used:

electrolyte: A2 of Struers

temperature of the electrolyte: RT

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

voltage: 25 V

- Final thinning using the ion milling facility (e.g. PIPS) at 3.5 keV, 4deg, time of the thinning should be adjusted to the specimen thickness.

Specimen (thin foil) is ready for TEM investigations.