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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

KRA (AGH University of Science and Technology) input to Deliverable 6.2:

Report on protocol for TEM sample preparation of Ni-base superalloys

TEM sample preparation of Ni-base superalloys

Nickel based superalloys exhibit very high strength at high temperatures, creep and oxidation resistance. Therefore, they are widely used in extreme environments, mainly in aerospace industry (parts of jet engine's e.g. turbine blades) and power generation (e.g. stationary gas turbines).
As in case of all materials, TEM specimen preparation of Ni base superalloys is a key issue for reliable results of microstructural investigations. The technique of preparation should not affect the microstructural features that will be analyzed. Specimen preparation should enable to achieve a representative part of a large specimen (e.g. after mechanical tests) in a form of a TEM specimen which will be observed and analyzed.

Steps of TEM specimen preparation of a nickel based superbased alloys:

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

- Initial thinning to the thickness of about 100μm,
- Cutting of a 3 mm disc,
- Final thinning: electropolisching

Fig. 1, Received piece of material

In the first step, slice(s) with the thickness of about 1 mm should be cut. It can be made with circular saw. During process of cutting the specimen and cutting disc should be cooled.

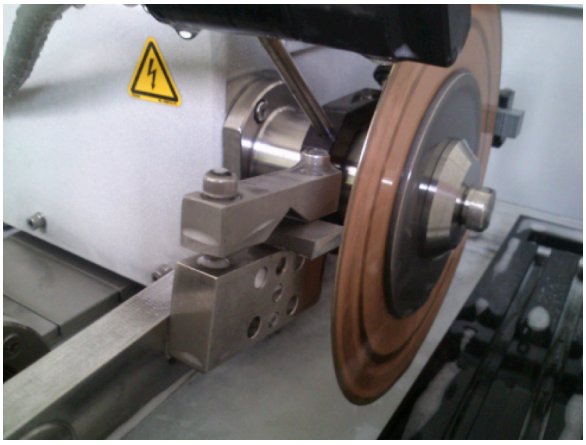

Fig. 2, Cutting of a slice of about 1mm thickness

In the next step specimen should be mechanically thinned to a thickness of max. 100μm. A special disc grinder (Fig. 3) which allows to control remaining thickness of the specimen may be used.

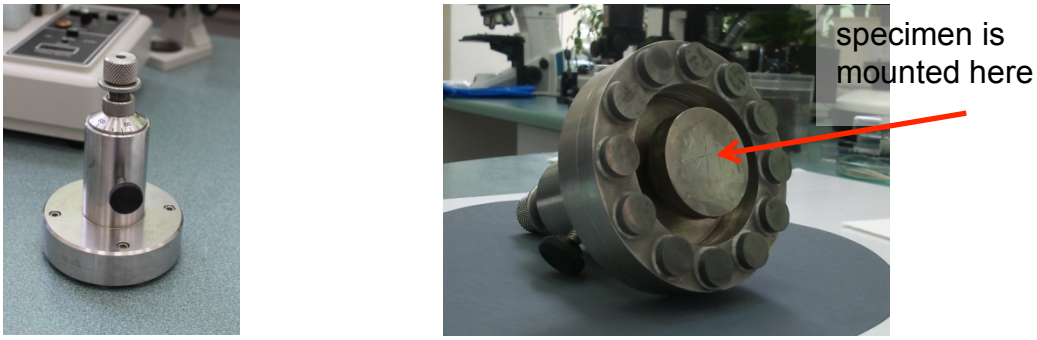

Fig. 3, Disc grinder, which allows to control remaining thickness of the specimen

The specimen should be glued to the disc grinder with a thermoplastic glue, which allows to unglue the specimen with an easy way.

Then the specimen should be grinded on a sandpapers, starting from about 400, through 600, 800, 1000 and finishing on 1200 grit.

In the following step, the specimen should be unglued, cleaned with acetone (in order to remove remaining of the glue). Then the thickness should be measured and the second side of the specimen grinded, again, starting from 400 till the sandpaper 1200 grid. The remaining thickness of the slice should be less than 100μm. After that the specimen should be unglued, cleaned with acetone and ethanol.

I the next step a 3mm disc should be cut. It should be made with a disc puncher.

Fig. 4, 3mm disc puncher

The last step of 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 (Fig. 5) 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 shuts off the process of electropolishing.

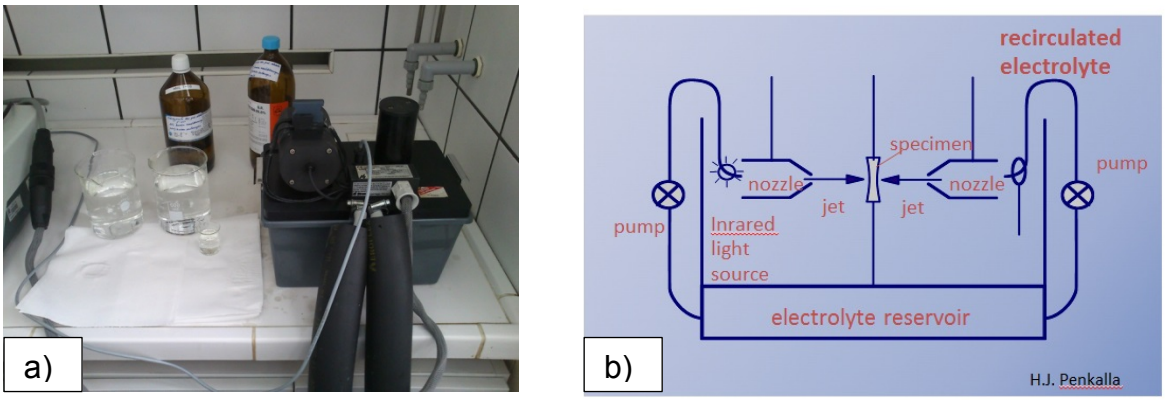

Fig. 5. Electropolishing unit (a), and a scheme of a twin-jet electropolishing apparatus (b).

During final thinning of a nickel based superalloys (using Tenupol 5) the following conditions may be used:

electrolyte: 10% perchloric acid and 90% acetic acid

temperature of the electrolyte: 10 ºC

flow rate: 10 (arbitrary unit)

voltage: 27 V

After process of electropolishing the specimen should be carefully cleaned in ethanol and distillated water.
Finally the specimen is ready for TEM investigations.

Safety issues

You should read information about solutions used for specimen preparation, always use protective cloths, gloves and eyeglasses.
Perchloric acid used during nickel based superalloys TEM specimen preparation is a very strong oxidizing agent and may be subject to local regulations. It is highly reactive with metals and organic matter (e.g. wood, plastics). However aqueous solutions (up to approximately 70%) are generally safe. Work conducted with perchloric acid must be conducted in fume hoods with a wash-down capability to prevent accumulation of oxidisers in the ductwork.
Concentrated **acetic acid** is corrosive to skin and must, therefore, be handled with appropriate care. Latex gloves offer no protection, so specially resistant gloves, (made of nitrile rubber) should be used during specimen preparation.