Deformation behaviour in Ni-based superalloys revealed by 4D-STEM crystal orientation analysis

Introduction

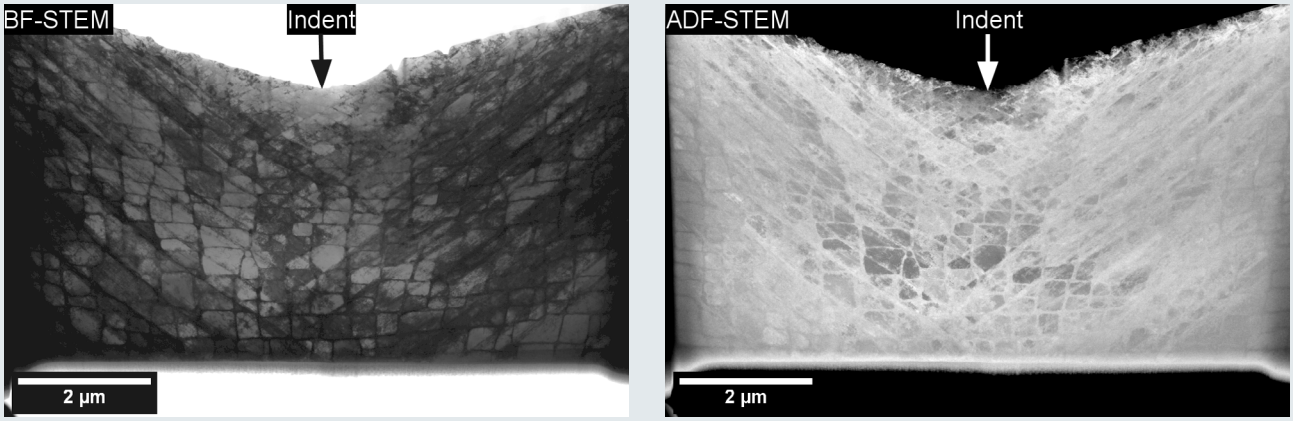
Nickel superalloys are advanced engineering materials for applications in demanding environments, including aerospace and energy generation, where they are subject to oxidising conditions, extreme operating temperatures up to 1000 °C, and complex mechanical stress states. Significant metallurgical research coupled with novel processing techniques have allowed the formation of single crystal gas turbine blades, with improved mechanical and creep resistance when compared to polycrystalline alloys. However, as they possess a more complex composition with multiple alloying elements, their deformation, creep, and fatigue behaviour must be thoroughly investigated across multiple length scales and operating conditions. As such, understanding the mechanical properties of these alloys will allow for effective and cost-efficient material selection.

This application note demonstrates an investigation into the crystallographic grain reorientation caused by Vickers indentation induced plastic deformation, studied by precession-assisted 4D-STEM with automated crystal orientation analysis.

The TESCAN TENSOR analytical 4D-STEM makes use of automatically generated kinematic diffraction templates to map changes in crystal orientation down to the few nm level, with a dedicated crystal orientation measurement. This measurement leverages fast kHz precession and a synchronised direct electron detector to enable rapid and efficient acquisitions of large datasets, with on-the-fly template matching to show first results whilst the acquisition is still running.

Methods

A Vickers indentation hardness test was performed along the [001] direction of a single crystalline Ni-based superalloy specimen with the nominal composition Ni-6.5Cr-9.0Co-0.6Mo-6.0W-5.6Al-1.0Ti-9.0Ta (wt%). Following this test, FIB lamellae parallel to the (100) plane were taken from the plastically deformed region under the



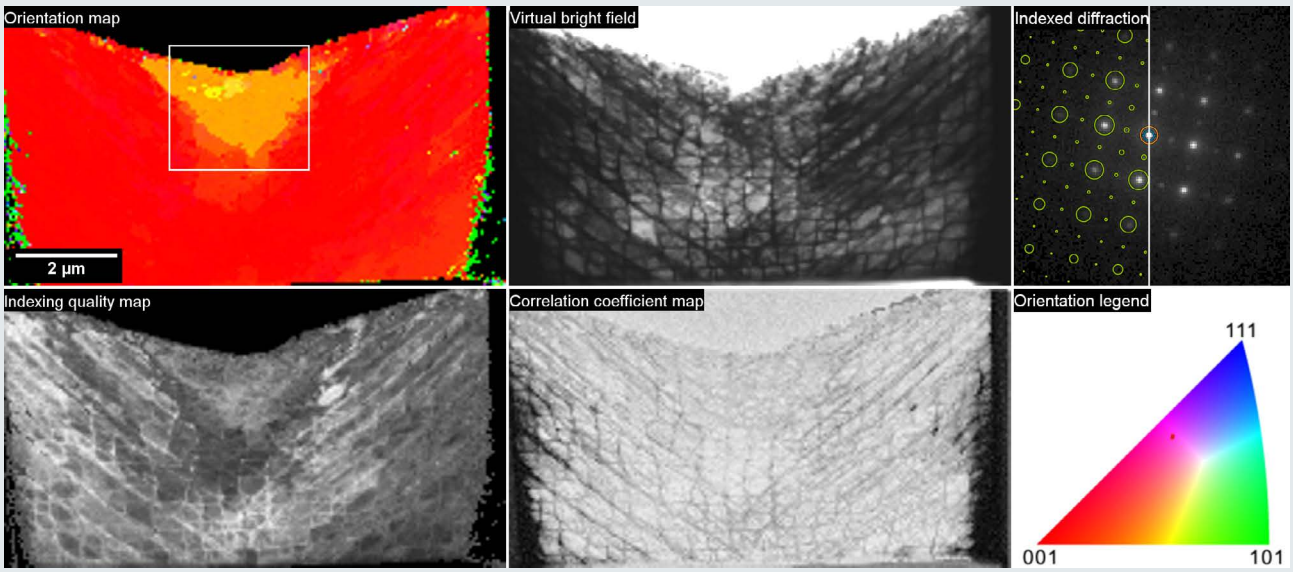
▲ **Figure 1:** BF and ADF-STEM imaging of the Ni-based superalloy lamella, showing cuboid grains and slip planes.

indent. These lamellae were then analysed with STEM imaging and precession assisted 4D-STEM with automated crystal orientation mapping. For STEM imaging, a 6.5 mrad convergence semi-angle and 100 pA probe current was used. For 4D-STEM orientation analysis, a 2 mrad convergence semi-angle was employed with a 50 pA probe current. The probe dwell time was set to 1 ms (1000 frames per second), with a pixel size of 55 nm for the overview map and 6.5 nm for the detail map, and precession angle of 14 mrad. Kinematic diffraction templates were generated for the known cubic structure of Ni_3Al with lattice parameter a = 3.56 Å and Pm-3m space group.

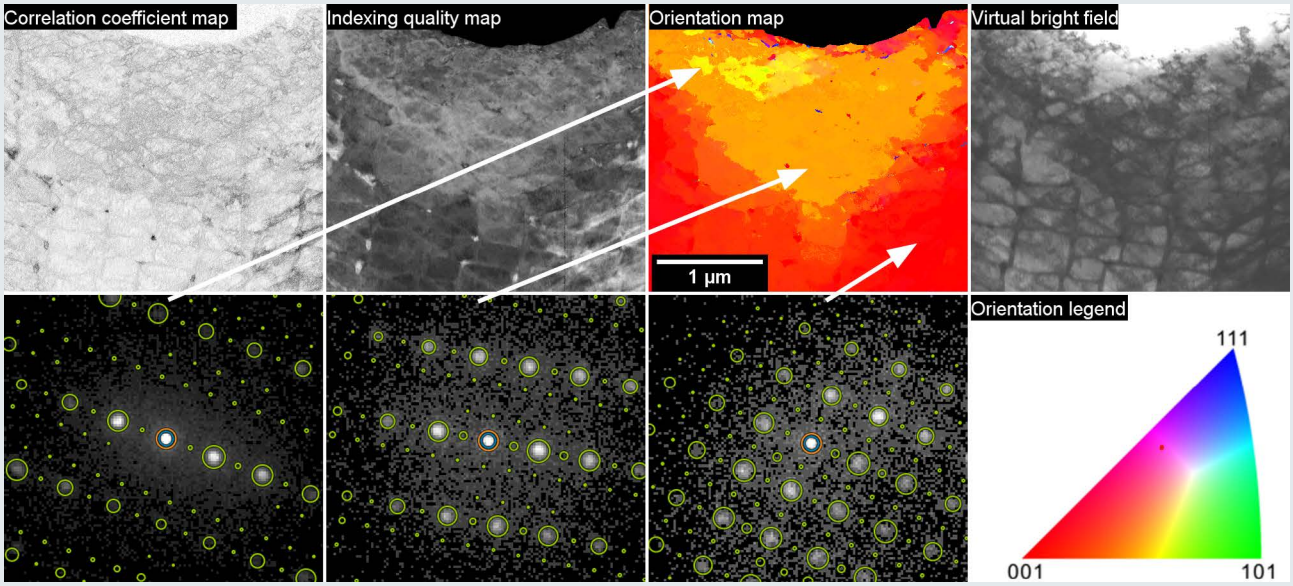
Results and Discussion

STEM imaging (shown in **Figure 1**) of the lamella demonstrates the presence of slip planes in the compression zone of the indent, proving heavy deformation under the point of the indent. From the STEM images, the presence of the two phases are easily distinguished. In Ni-based superalloys, these phases are known as face-centered cubic (FCC) γ framework and $L1_2$ structured γ' cuboids, and thus they appear as narrow γ-channels and γ'-rectangles in the STEM thin section. Prior to indentation, both γ and γ' were crystallographically aligned obeying the cube-cube orientation relationship and the specimen produces single crystal diffraction patterns. Spots with dark contrast were also observed underneath the indent, which will be discussed in detail in later sections.

An overview orientation map acquired from the whole area of the thinned lamella **(Figure 2)** was used for checking the diffraction pattern quality, localising the region of interest, and refining the diffraction template processing parameters. On-the-fly processing of the orientation analysis data meant that regions suitable for further study could be rapidly narrowed down, vastly increasing microscope time efficiency compared to traditional offline processing. Based on the results provided by the overview map, a region with large orientation gradient right under the indentation point (marked by the white box) was selected for a high spatial resolution orientation measurement. This measurement and processing refinement were performed within the same microscope session, though more detailed processing refinements could be undertaken offline after acquisition also.



▲ **Figure 2:** Whole lamella orientation mapping with virtual bright field, indexing quality map, correlation coefficient map, colour legend and template matched diffraction pattern showing both the γ (weaker spots) and γ' phases are crystallographically aligned.



▲ **Figure 3:** High resolution orientation map with correlation coefficient map, virtual bright field image and orientation legend, indexed diffraction patterns are taken from the points indicated with arrows.

The higher resolution orientation map **(Figure 3)** showed that the areas of heavy slip plane density (the dark lines angled at 45 degrees in the virtual bright field image) were correlated with areas showing change in orientation. Regions between parallel slip planes can form kink bands, where the crystal rotate with respect to the undeformed state. The change in orientation in the orange region from [001] to [103] showed high indexing quality due to the good diffraction template matching (shown by brighter regions in the indexing quality map). Within the deformed orange region, a subregion, shown in yellow, displayed a larger orientation change but relatively poor indexing quality. This was likely because the region directly under the indent underwent the largest plastic deformation and thus the quality of the diffraction patterns was low, although it can be seen that the diffraction templates still matched relatively well.

Conclusions

Automated crystal orientation analysis using the TESCAN TENSOR provided novel insights into the deformation behaviours of Ni-based superalloys. From the results presented, the indentation deformation induced crystallographic orientation gradient was mapped with high resolution. STEM imaging clearly demonstrated the presence of slip planes, which correlated well to the crystal orientation changes.

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