Identification of intermetallic σ-phase in annealed 316L

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Introduction & Problem

MYRRHA (Multi purpose hYbrid Research Reactor for High tech Applications) is an accelerator driven system, currently under development at SCK CEN in Mol, Belgium. Within the MYRRHA materials program, candidate materials for the **structural parts** of this new generation reactor are investigated, including 316L-type steels.

A specific batch of solution annealed 316L-type steel with high Cr and Ni content showed **unexpected mechanical behavior** (Fig. 1) when tested in the through-thickness orientation. The fracture surfaces of these specimens showed untypical signs of brittle behavior (Fig. 2). This triggered further investigations in order to identify the origins of this behavior.

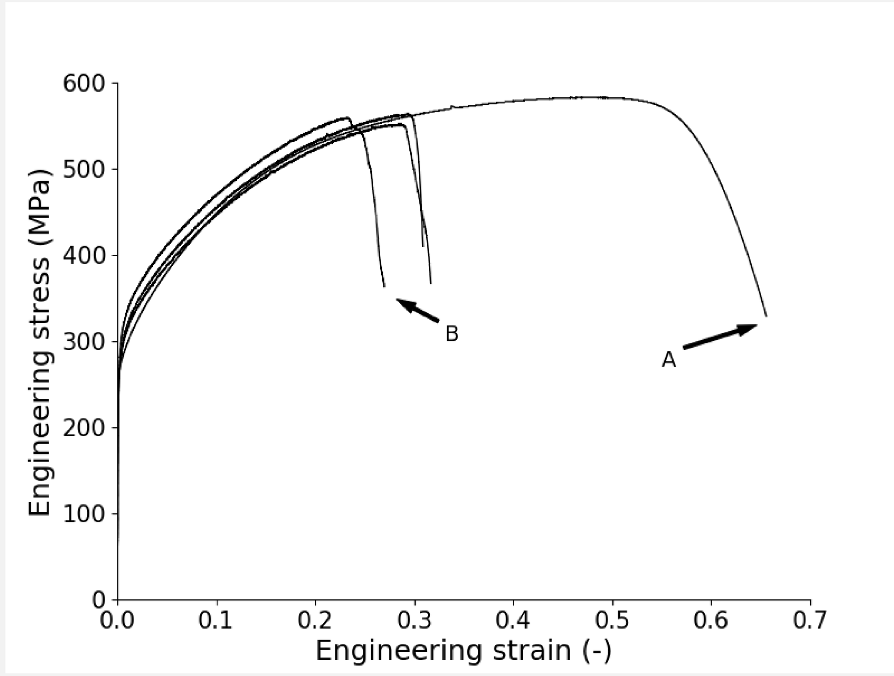

Fig. 1: Engineering stress-strain curves of uniaxial tensile tests where specimens are extracted in the through-thickness (S) orientation, tested in air at room temperature with a strain rate of $5 \cdot 10^{-5}$ s^{-1}.

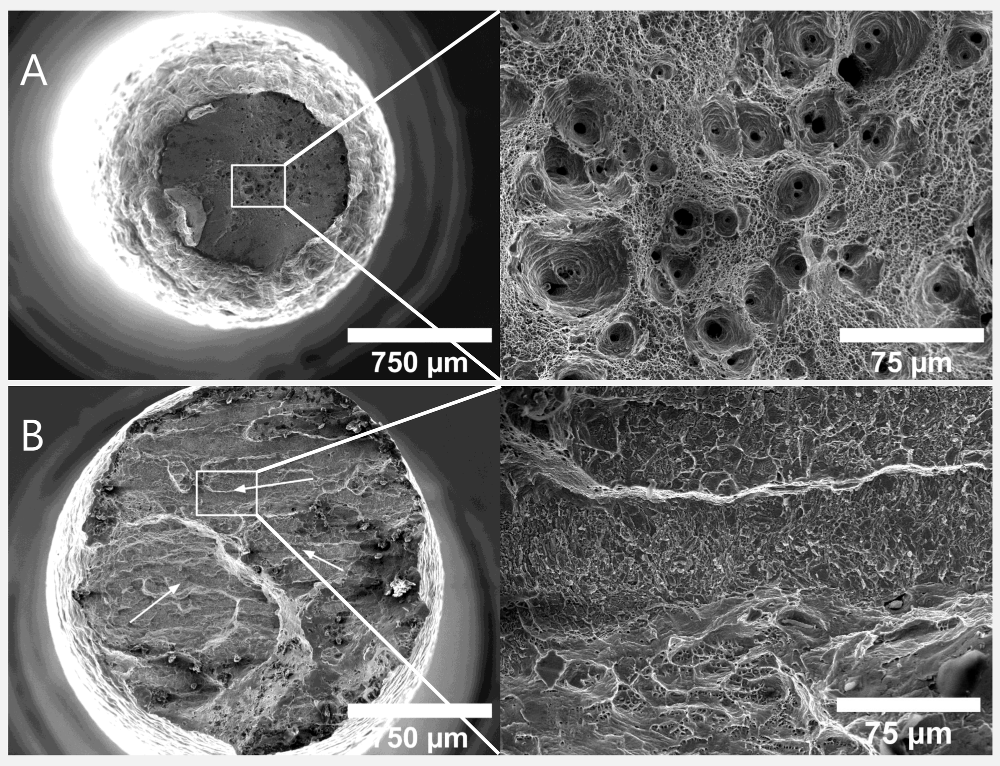

Fig. 2: SEM images of fracture surfaces of tests A and B shown in Fig. 1.

Results

Could it be δ-ferrite?

Delta-ferrite is common in 316L and expected. When measuring the magnetic response of this material, the fraction of this ferromagnetic phase can be determined, and a discrepancy is noticed when compared to the optical analysis of the phase fraction of the supposed δ-ferrite (Fig. 3). **Something is missing…**

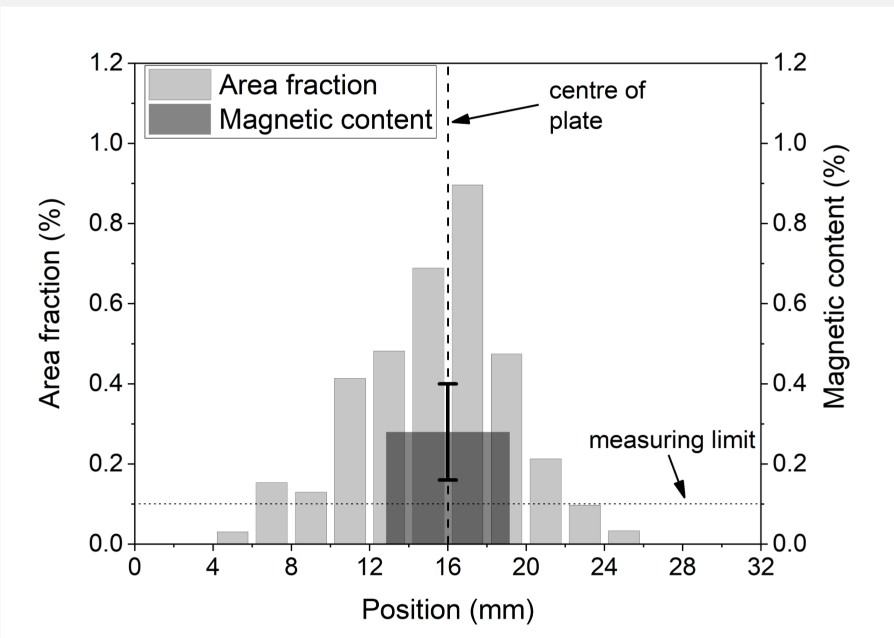

Fig. 3: Comparison of the measured area fraction of stringers (measured by optical microscopy) and δ-ferrite content (measured by Feritescope)

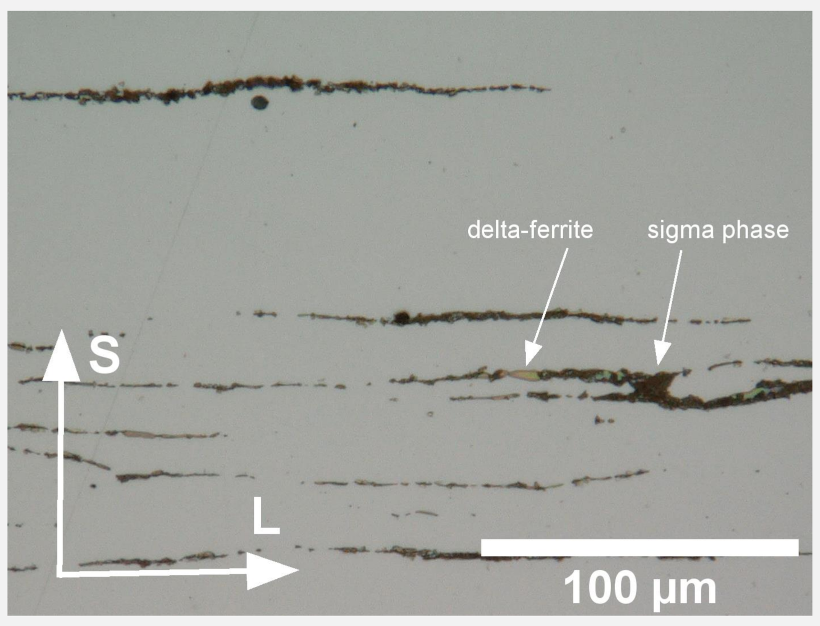

Fig. 4: Optical images after etching (Murakami etchant) of center of plate

Could it be intermetallic σ-phase?

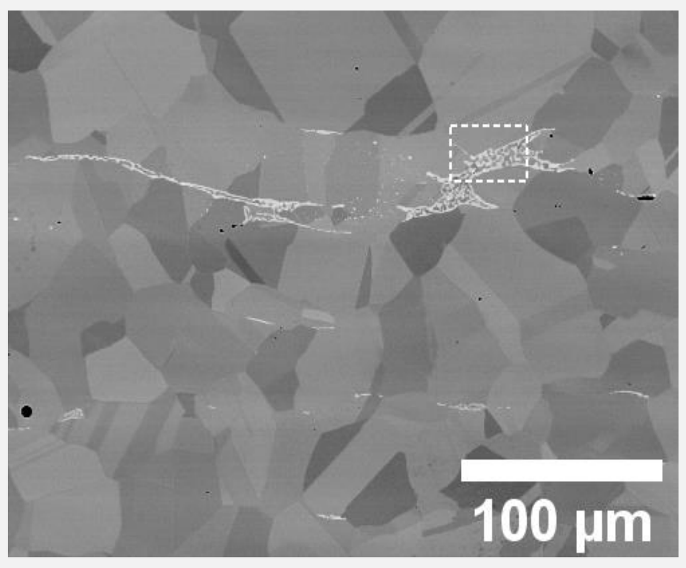

Fig. 5: SEM image using backscattered electrons signal from a zone with a high density of intermetallic phase.

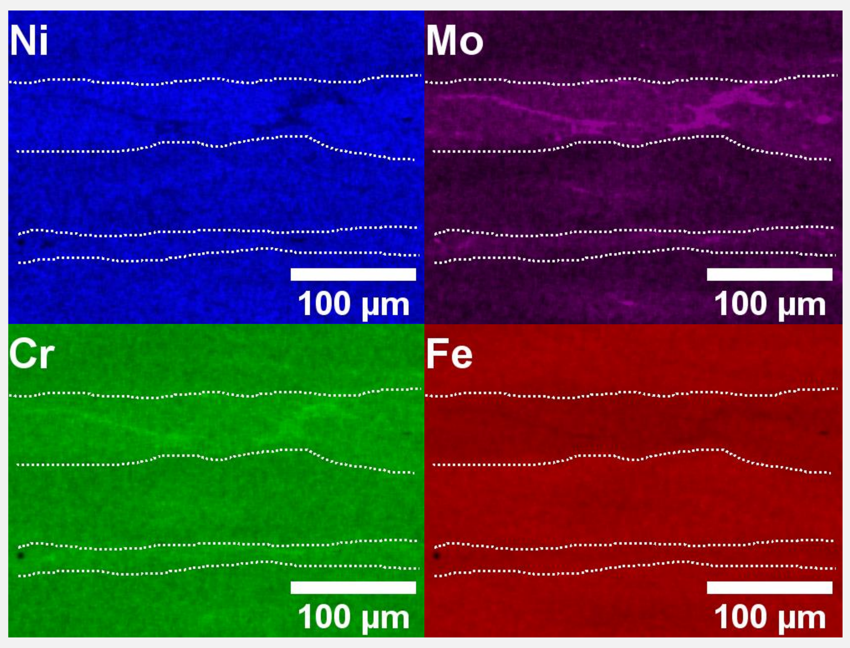

Fig. 6: EDX chemical mapping of region shown in Fig. 5

- EDX measurements showed high-Cr (25%), high-Mo (7%) content, consistent with σ-phase and local micro-segregation
- EBSD mapping showed low-quality Kikuchi-pattern. Comparison with kinematically simulated patterns showed good agreement with intermetallic σ-phase
- EBSD identification of the Laves phase was incorrect, and originated from a superposition of diffraction signal from neighboring austenitic grains

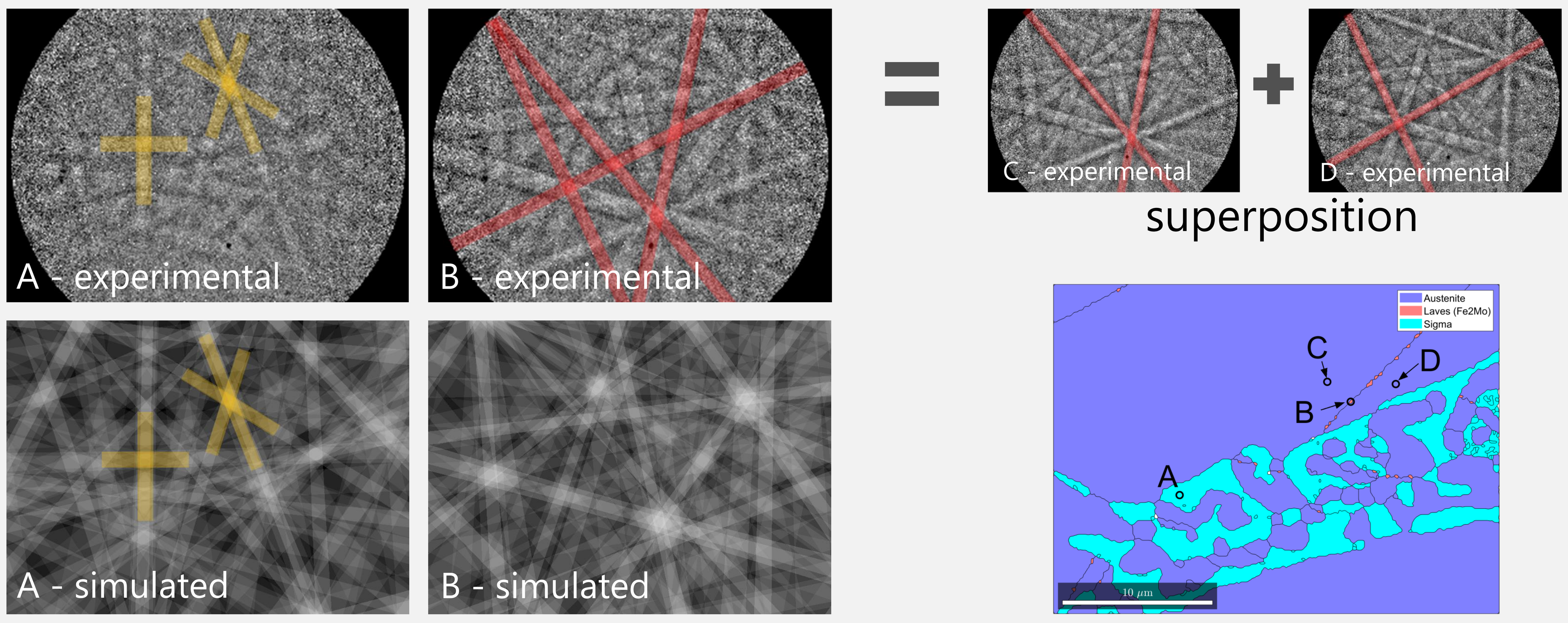

Fig. 7: EBSD phase map of indicated zone in Fig. 5 and corresponding experimental Kikuchi patterns recorded on different positions. According to the identification by the software at position A and B, phase and orientation were used to generate kinematically simulated patterns. Position A was correctly identified as σ-phase, position B was wrongfully identified as Laves phase.

Conclusion

Successful identification of unexpected intermetallic σ-phase

- Intermetallic σ-phase was detected in a solution annealed 316L plate by combining mechanical testing and in-depth microstructural analysis
- Simulated EBSD diffraction patterns were successfully used to validate low quality indexations of different phases
- Additions to the standard qualification procedures of steels containing high amounts of Cr and Mo are recommended

More information?
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