A TEM investigation of the influence of nitrogen and cold working on the microstructure of solid solution annealed austenitic stainless steel (316L)

Introduction

The purpose of this study was to investigate the influence of cold deformation and the N content on the stress corrosion cracking (SCC) of austenitic stainless steel (316L). It was previously discovered that the material with high N content was more susceptible to SCC. It was therefore suspected that the higher N content resulted in a larger amount of nitrides (containing Cr), which would deplete the matrix and/or the grain boundaries of Cr.
Two types of materials (high and low N content) were investigated, both in the as-received state (solid solution annealed) and after 33% cold deformation.
However, since the investigated materials had not been subjected to any heat treatment, no Cr nitrides could have formed, and the TEM investigation was focused on the influence of N on the number of precipitate nucleation sites.

Experimental

Four different materials (summarised in Table 1) were studied. Thin foil specimens were prepared for the TEM by electropolishing in 10 % perchloric acid in methanol at -20 °C.

Table 1 Investigated materials.

Designation	N content (wt%)	Cold working
A	0.049	0%
B	0.049	33%
C	0.17	0%
D	0.17	33%

One or two specimens of each material were investigated in a Philips CM200 Supertwin FEG-TEM, operated at 200 kV, and equipped with a Link ISIS energy dispersive X-ray spectrometry (EDS) unit.

Results

Very few precipitates were observed in the four materials. This is expected, since the materials had not been subjected to any heat treatment, during which precipitates could form. In a previous study, CaS and Laves phase precipitates were observed, but

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in this investigation, no such phases were found. One reason for this could be the limited specimen area (roughly 500 to 1000 µm^2) that is accessible in a TEM specimen. Hence, it can not be ruled out that such phases were present in the investigated materials, since such precipitates are expected to be fairly large and sparsely distributed.

The grain size of the undeformed materials (A and C) was approximately 5 to 15 µm (Fig. 3). In these materials, approximately 5 to 10 grain boundaries were accessible in each TEM specimen. Some twinned regions with dislocation bands were also found in material C (Fig. 4). EDS measurements across grain boundaries and twins did not reveal any segregation or depletion at these regions, as shown in the EDS profile in Fig. 1. This profile was taken from the area shown in Fig. 2. No clear texture was observed.

In the cold-worked materials (B and D), the dislocation density was very high and distinct twin laths were observed. These features obscured most of the initial grain boundaries. However, as seen in Fig. 5 and Fig. 6, the density of twin boundaries was much lower in the material with low N content (about 10/µm) compared with the material with high N content (about 30/µm).

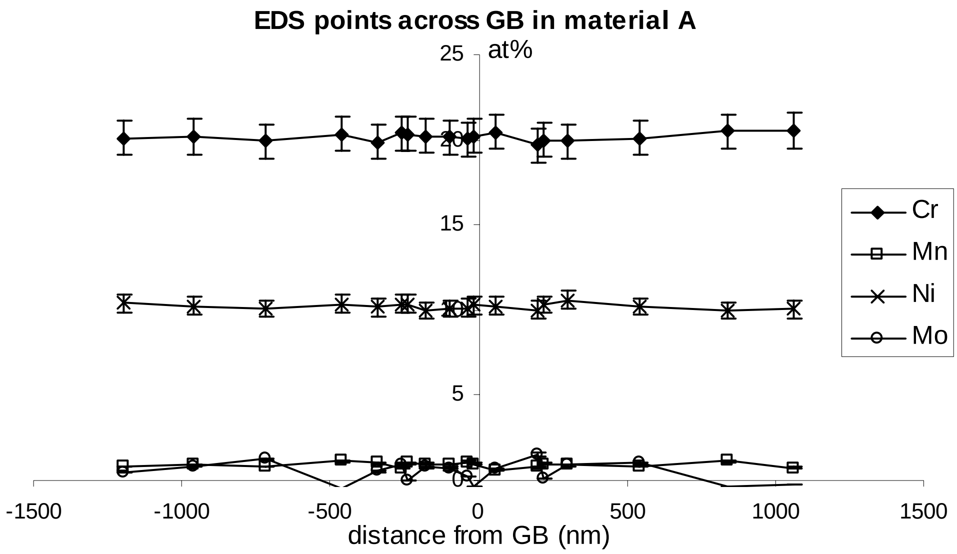

Fig. 1 STEM-EDS measurements across a grain boundary in material A. The effective spot size was approximately 2 nm. Close to the grain boundary, the step size was around 20 nm.

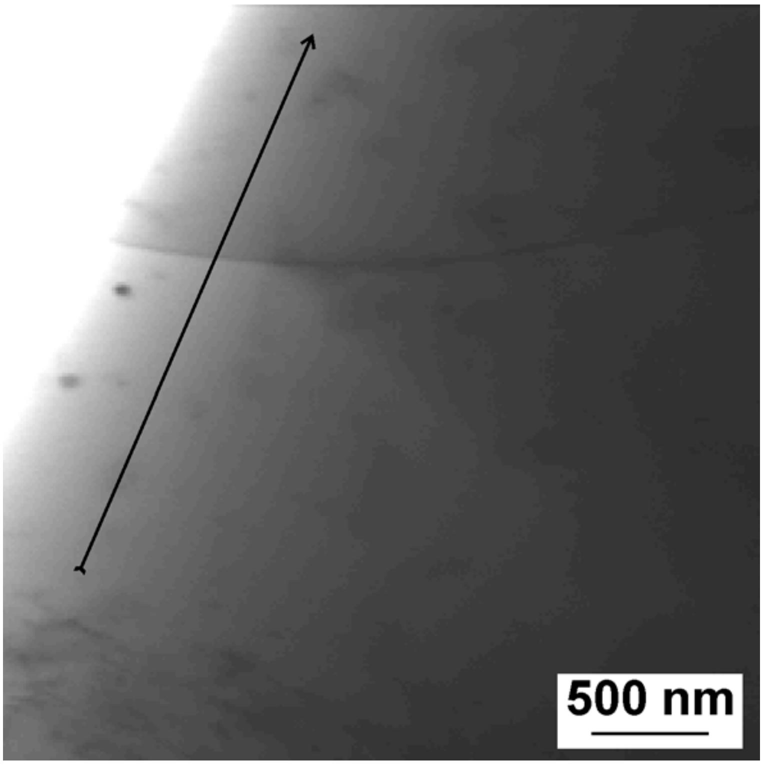

Fig. 2 STEM image, showing a grain boundary in material A. The arrow shows the EDS profile.

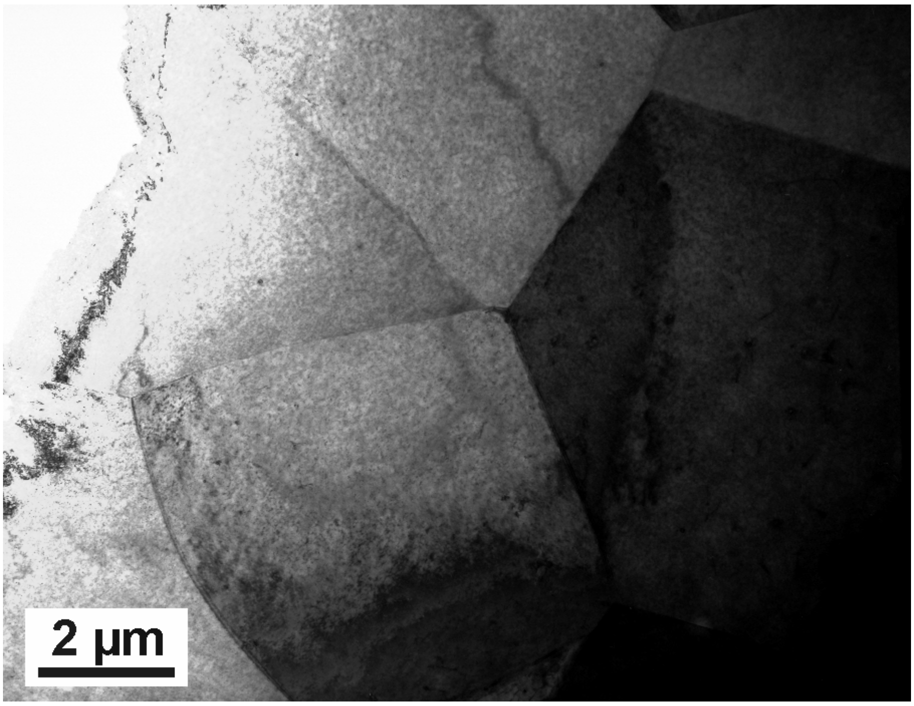

Fig. 3 Typical microstructure of the undeformed materials (material A).

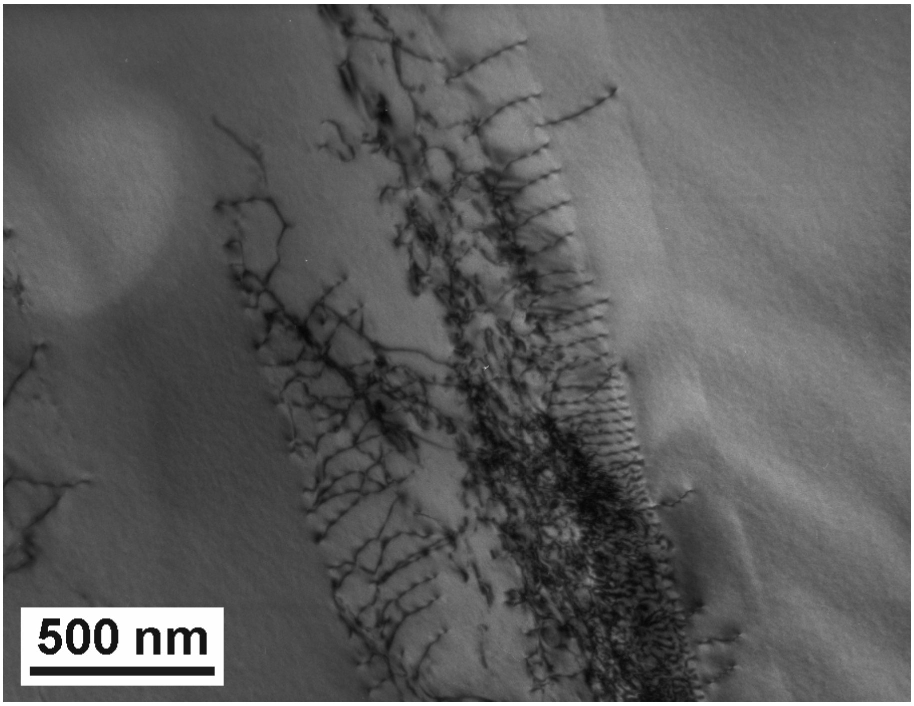

Fig. 4 Occasional dislocation bands were seen in material C.

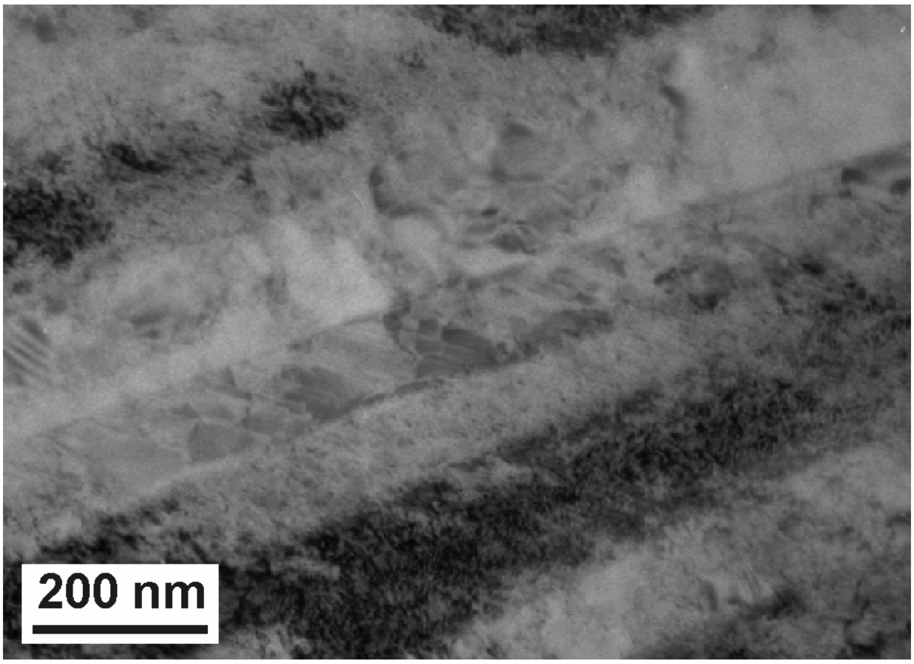

Fig. 5 Twin laths in the low N material after cold deformation (B).

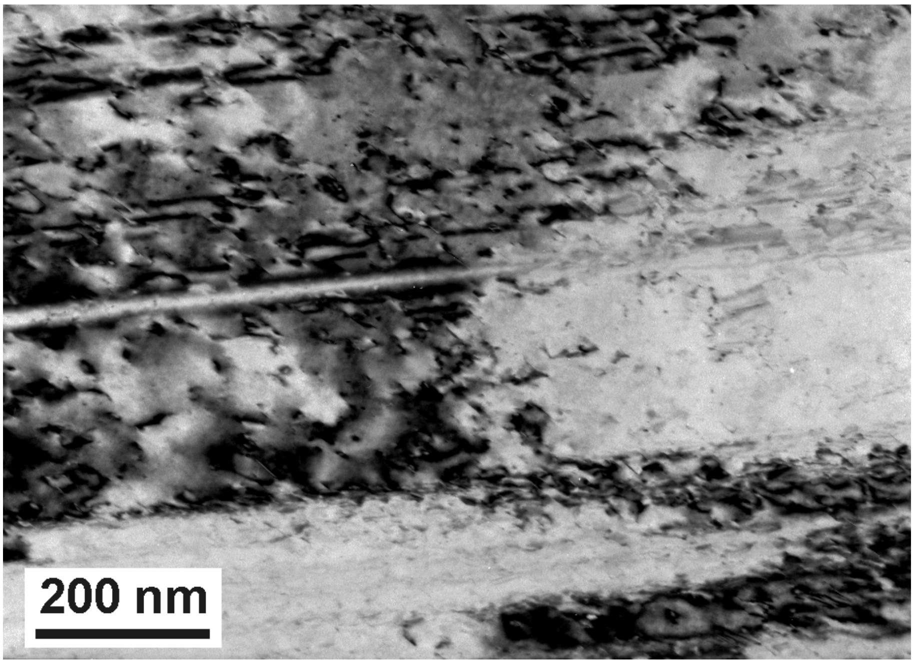

Fig. 6 Twin laths in the high N material after cold deformation (D).

Other observations

In material C (high N, undeformed) some occasional Y and Al- rich inclusions were also observed. A bright-field image of the Y-rich particle is shown in Fig. 7 and EDS spectra from the Y- rich and Al-rich particles are shown in Fig. 8 and Fig. 9, respectively. In both these spectra, it can be expected that there is some contribution from the surrounding matrix.

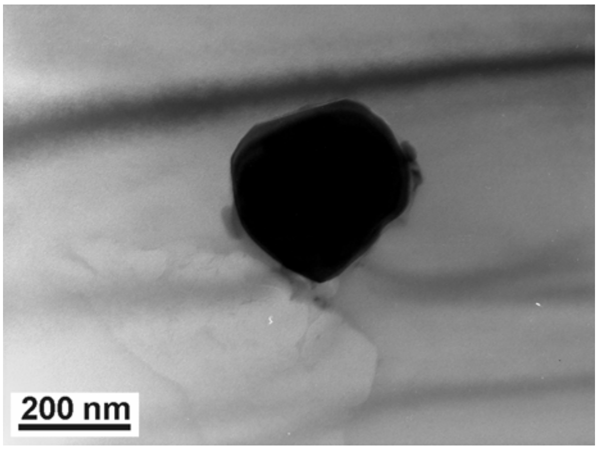

Fig. 7 Bright-field image of an Y-rich inclusion in material C.

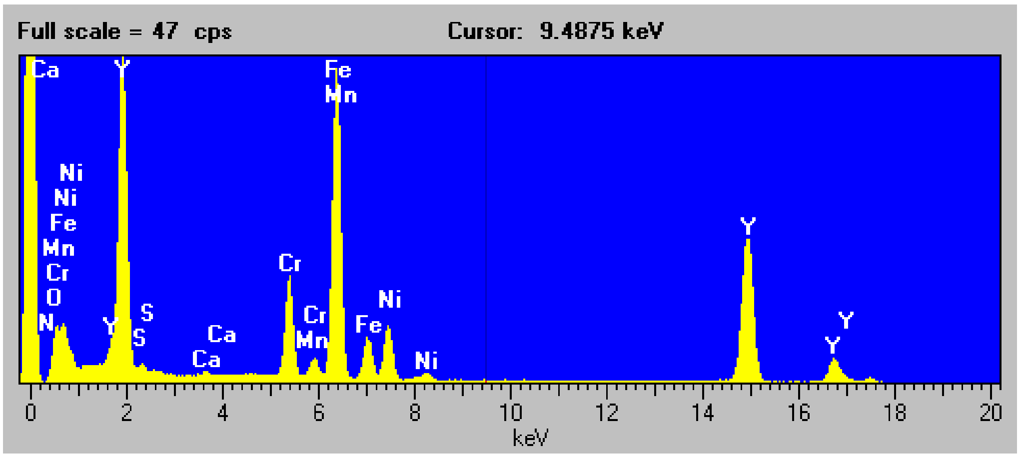

Fig. 8 EDS spectrum from an Y-rich inclusion in material C.

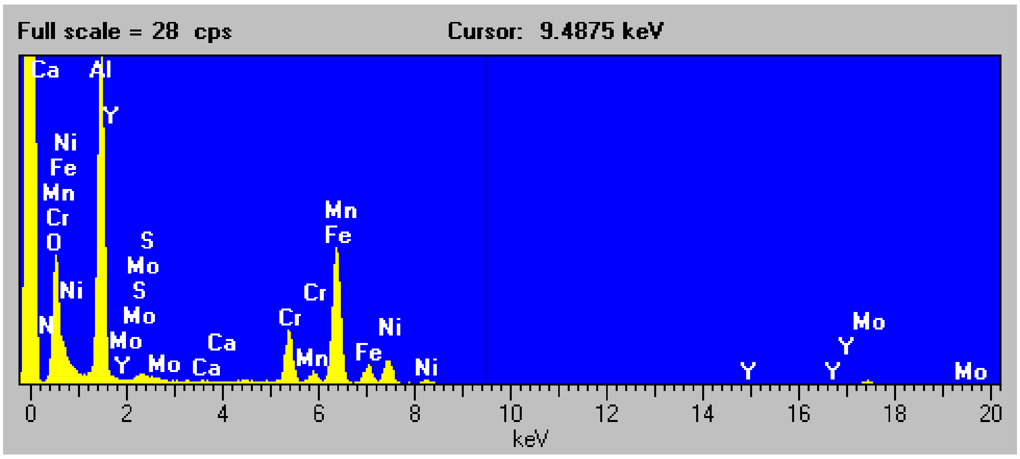

Fig. 9 EDS spectrum from an Al-rich inclusion in material C.

Discussion

As mentioned above, it is not expected that any precipitates (carbides or nitrides) should be present in these materials, since they were in the solution-annealed state. However, during heat treatment, the nucleation rate of precipitates is (besides the diffusion rate of the precipitate-forming species) controlled by the number of nucleation sites. Such sites may be: grain boundaries, dislocations, stacking faults etc. Naturally, the deformed materials in this study contain more dislocations and stacking faults. On the other hand, it also appears as if the number of grain boundaries was less in the deformed materials.

In the present study, it was observed that after cold deformation, the stacking fault density was higher in the material containing more N. This can be explained by the lowering of the stacking fault energy at increasing amounts of N [1]. Thus, it is expected that a higher amount of N increases the precipitate nucleation site density, which in turn increases the rate of depletion of Cr from the matrix during consequent heat treatment due to the incorporation of this element to the precipitates.

Additionally, it can be speculated that the higher amount of N should increase the amount of Cr nitrides if this phase forms during heat treatment. Any increased Cr-nitride precipitation should severely increase the Cr depletion and consequently also the susceptibility to stress corrosion cracking. However, to test this hypothesis, it is necessary to perform further TEM studies on materials that have been heat treated.

Summary and conclusions

- Since the materials still were in the solution-annealed state (besides the cold deformation), very few precipitates were observed in the studied materials.
- No Cr depletion at grain boundaries was observed in any of the materials.
- It was observed that a higher amount of N increased the density of stacking faults after cold deformation. This should increase the number of precipitate nucleation sites, which in turn should increase the rate of Cr depletion due to precipitation during consequent heat treatment.
- It was also proposed that the higher amount of N should increase the amount of Cr nitrides (if these form at the work temperature), which should increase the Cr depletion and the stress corrosion cracking susceptibility. To confirm this assumption, it is necessary to investigate materials that have been heat treated at a suitable temperature.

References

1. L. A. Norstrom, Met. Sci., 11, (1977) 208.