Dislocation substructure in \textit{in situ} deformed foils of niobium–8 to 10 at % vanadium alloy
Part 2

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The development of dislocation substructure in foils of niobium–8 to 10 at % vanadium alloy deformed \textit{in situ} in the tensile stage of the microscope is presented to substantiate earlier observations presented in Part 1. The results of direct observation of the formation of straight dislocations, dislocation loops and loop debris are presented. In addition, the behaviour of cracks in foils containing hydrogen is compared with that observed in the absence of the interstitial. Through the \textit{in situ} observations, the mechanism of embrittlement of niobium-rich vanadium alloys in the presence of hydrogen presented in Part 1 is substantiated.

1. Introduction
The development of dislocation substructure in the niobium–8 to 10 at % vanadium alloy has been described in Part 1 to determine the influence of hydrogen present in the interstitial sites in the lattice [1]. It has been demonstrated that cracks observed along active slip planes in the hydrogen-embrittled alloy single crystals are a result of early formation of dislocation cell walls. Further, accumulation of hydrogen along the cell walls leads to easier crack nucleation. On the other hand, the presence of straight long screw dislocations and edge segments is attributed to the effect of alloying by substitutional vanadium. In order to verify the above conclusions, foils of this alloy have been deformed \textit{in situ} in the electron microscope and the results are presented in this paper.

Recent direct observation of thin foils of iron, of thickness less than 1 \(\mu\)m, deformed \textit{in situ} in the tensile stage in an electron microscope equipped with an environmental cell has shown that cracks already present in the foils under constant tensile load moved due to the enhanced mobility of dislocations arising upon introducing hydrogen [2, 3], a solid softening effect. However, other authors [4] propose that the growing and changing concentration of hydrogen atmosphere drags the dislocations towards regions of the crack tip. On the other hand, enhanced dislocation motion is observed as an independent effect upon introducing hydrogen into the foils containing no cracks [2]. Yet another important and more direct local effect near the core of the dislocation will be the additional plastic strain introduced when hydrogen atoms accumulate and as a result give rise to tetragonal distortion associated with the interstitial. In fact, this effect similar to others will be proportional to the partial pressure of hydrogen at which it is introduced into the environmental cell, a result observed experimentally [2, 3] with the applied specimen displacement held constant. Further support to this contention is obtained from the fact that either helium or air did not have a similar effect since their diffusion into the foil is doubtful. In addition, the high mobility of hydrogen atoms and their interaction with the core of the screw dislocations can be responsible for their movement. Therefore, the observations of either dislocation movement or crack growth upon introduction of hydrogen with the foils under constraint may not be applicable to the situation when prior hydrogen charging is done. We do not intend to underscore the importance of charging the specimen under load in the environmental cell with hydrogen, since this may correspond to the actual situation encountered. On the other hand, it is our aim to emphasize the difference between the two categories of experiments, in particular when the solubility of hydrogen in iron is considered to be small [5].

In this respect, \textit{in situ} experiments carried out on the niobium–8 to 10 at % vanadium alloy possessing a large interstitial solid solubility of hydrogen should be useful in understanding the effect of hydrogen. The trapping of hydrogen atoms to vanadium arising from either the electronic or the strain field interaction [6], which is responsible for the high hydrogen solid solubility in this alloy, is considered to prevent the hydrogen atoms from diffusing out of the foils tested in vacuum. In fact, it has been found that degassing of the previously charged specimens from hydrogen at \(10^{-7}\)torr vacuum required raising the temperature above 400 to 500°C illustrating the tendency for hydrogen to remain within the specimens. The important parameter that seems to determine the nature of results obtained in the \textit{in situ} experiments is the thickness of the foil [7]. It has been found that the very ductile metals show a brittle behaviour when the foil...
thickness is reduced below a certain limit. In this respect, the experiments performed on the niobium–vanadium alloy have comparable thickness when tested either with or without hydrogen. The operating voltage of the Jeol 100 CX electron microscope, in which the present experiments are carried out, is limited to 120 kV and as a result, the thickness of the foil that remained electron transparent is between 0.2 and 0.5 μm. In addition to the plane stress conditions in which the planes of maximum resolved shear stress different from those in plane strain, the constraints imposed by the free surfaces on the dislocation mobility can give rise to different results from those observed in the macroscopic specimens. On the other hand, the present observations will be useful in understanding the development of dislocation substructure and cracks in the presence of hydrogen at room temperature.

2. Experimental procedure
Specimens of niobium–8 to 10 at% vanadium alloy that can be pin loaded in tension, 8 mm × 3 mm × 1 mm in dimension were spark cut from a rolled foil. Further, the specimens were ground to 500 μm thickness, first on 320 grit followed by 600 grit silicon carbide papers and further electropolished to provide a smooth surface using an electrolyte of 6 vol% sulphuric acid in methanol. In order to start with an initially dislocation-free foil, the specimens were annealed for about 10 h at 1400°C under 10⁻⁷ torr vacuum. On the other hand, charging with hydrogen is done by taking the foils to 950°C at 10⁻⁶ torr vacuum and after isolating the furnace from the vacuum system, a premeasured volume of hydrogen from decomposition of uranium hydride is introduced into the furnace. The charging procedure is completed by keeping the specimens in a hydrogen atmosphere for 3 h and cooling the furnace. In addition, an electron transparent area of the foil is obtained in the centre of the specimen in the manner already mentioned in Part 1. The side entry eucentric stage, capable of single tilt about its axis by ± 55° and displacement controlled is used so that strain can be interrupted at any instant to record the observations. At the same time, a video camera has been placed in the front port and changes in the dislocation substructure as well as crack-related structure are recorded using a VCR. Instead of continuous loading of the foil, incremental elongation is carried out and between each increment, the structure is allowed to come to equilibrium. Several foils, both free of hydrogen and those containing hydrogen, have been deformed in situ in the transmission electron microscope. Some characteristic features of dislocation substructures observed are presented in this paper.

3. Initial substructure of the foils
The substructure in the annealed foils is relatively free from dislocations. On the other hand, all the dislocations present in the foil have rearranged in the form of sub-boundaries during annealing, as shown in Figs 1a and b. The structure of sub-boundaries changed its regularity whenever a lattice dislocation joined the boundary. In particular, the continuation of the lattice dislocation as part of a boundary dislocation should necessarily imply that the boundary is fairly coalesced [8]. These substructural features are not easily observed upon straining the foils by only a little. In other words, the lattice dislocations associated with the boundaries are found to be active sources provided the applied stress has a suitable component.

4. Dislocation substructure and slip line observations
The formation and development of slip lines and the associated dislocation lines is illustrated in Figs 2a and b as a function of increasing strain. In particular, the straight slip lines initially formed (Fig. 2a) increased in density and more were generated with increasing strain. In addition, the expanding dislocation configuration shown in Fig. 2b is similar to the observations presented in Part 1 (Fig. 3b). The fully developed straight dislocations in the top and the expanding dislocation configurations at several places in the composite picture (Fig. 2b) illustrate very clearly the higher lattice frictional stress resulting from the substitutional alloying of niobium with vanadium at room temperature. A schematic illustration of the expanding dislocations and the formation of straight screw dislocations in the foil subjected to tension, as shown in Fig. 2b is given in Fig. 2c. The shear stress component on the edge segments is responsible for the growing dislocations on each slip plane. The movement of dislocations schematically described in Fig. 2c is also illustrated in the micrographs shown in Figs 3a and b in which no cross slip is observed but extensive loop and debris formation is noticed (Fig. 3b). Furthermore, dislocations of opposite sign moving in the opposite direction can be seen in Fig. 3b. Thus, the left-hand side of the picture shows the dislocations moving up while the dislocations on the right are moving down; with the nature of the dark and light shades of contrast also reversed. The formation of loop debris in these foils and possibly in the macroscopic specimens could arise as a result of combination of positive and negative segments from the expanding dislocations in the opposite direction. A more direct evidence of this mechanism is seen in the dislocation substructure associated with a crack tip in a virgin crystal shown in Figs 4a and b. In addition, some moving segments curve around into a loop-like configuration and join together with another adjacent dislocation to form a complete loop. The dark and bright lobes of contrast associated with each terminating screw dislocation at the surface after completion of the loop formation can be seen [9]. These observations substantiate earlier conclusions of Part 1, on the effect of substitutional vanadium in increasing the lattice frictional stress to movement of both edge and screw dislocations.

5. Dislocation substructure associated with cracks
5.1. Foils containing no hydrogen
It has been pointed out earlier that the dislocation substructures observed in thinner foils are distinctly