TEM and SEM investigation of nanostructure parameters of nuclear reactor pressure vessels

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Since the degradation of the mechanical properties of nuclear reactor pressure vessels (RPV) under irradiation and long thermal ageing is stipulated by formation of radiation defects (dislocation loops), nano-size precipitates, intragranular and grain-boundary impurity segregations, obtaining of the direct experimental data about principles of nanostructure evolution through all operation stages is an important experimental task. Such data is also actual because up to nowadays there is large number of theoretical studies but still no integral adequate model of radiation embrittlement that could explain and predict the changes of mechanical properties of RPV steels during irradiation and annealing.

In this work the dark-field transmission electron microscopy is used for visualization of nano-size coherent inclusions and precipitates. Quantitative measurements of the fraction of brittle (fig.1a) and ductile intragranular (fig.1b) fractures on the fracture surfaces SEM-images of samples after bending tests for determination of development of grain-boundary and intragranular segregation processes during long-terming irradiation at operation temperatures typical for reactor pressure vessel steels are suggested.

The first problem of nano-size precipitates studying in RPV steels is the sample-induced magnetic field influence on microscopy resolution in TEM-mode. The second one is that precipitates due to their small size are coherent with matrix and therefore do not form their own diffraction pattern. Therefore it is very difficult to investigate matrix-coherent inclusions and precipitates by means of classic dark-field and bright-field electron microscopy techniques.

For the first problem solution i.e. for minimization of objective lens spherical aberration the so-called method of adjusted dark-field image is suggested. It consists in complete adjustment of electron beam and microscope optical axis alignment directly within the grain of the magnetic material being studied. The midpoint of this procedure is the Volt center adjustment under conditions when electron beam pass directly through the studied area of the sample.

For the second problem solution the method of weak beam dark-field images is applied. The core of this method is in visualization of matrix crystal lattice distortion at the coherent inclusion area in conditions when the matrix reciprocal lattice point is displaced a little bit from Bragg position and accordingly the formation of strong diffracted beam doesn’t happen. This way the conditions when in reflective position are only distorted areas of matrix lattice, i.e. areas with precipitates (fig 2a) can be obtained. It is well known that the weak beam method also allows the significant enhancement of resolution under dislocation and dislocation loops images formation (fig. 2b). Since displacement of matrix lattice point from Bragg position under weak beam images formation is made by tilting the beam, the important thing is to avoid electron beam displacement during beam tilting on the selected area of the sample.

This paper shows that systematic combined TEM and SEM studies of RPV steels allow monitoring of their condition and help prediction of residual radiation resource [1,2].

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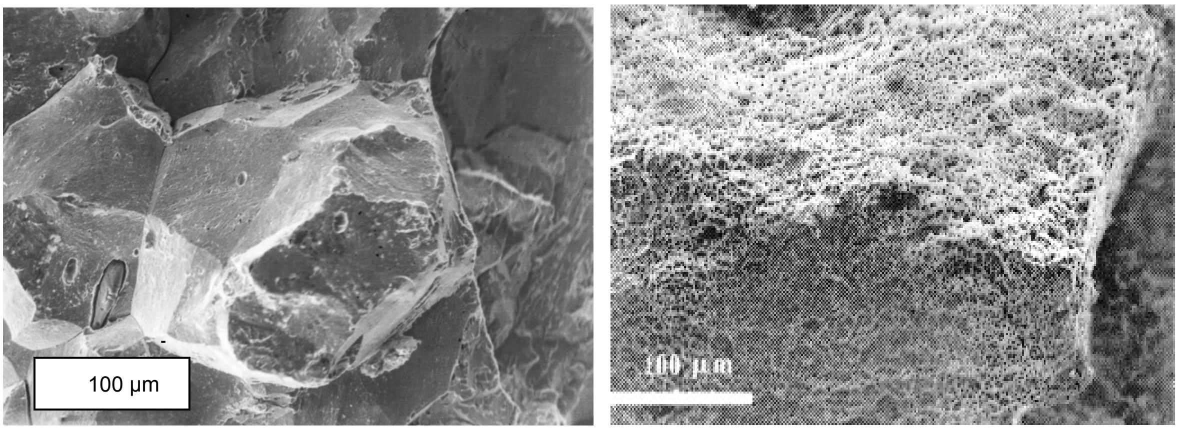
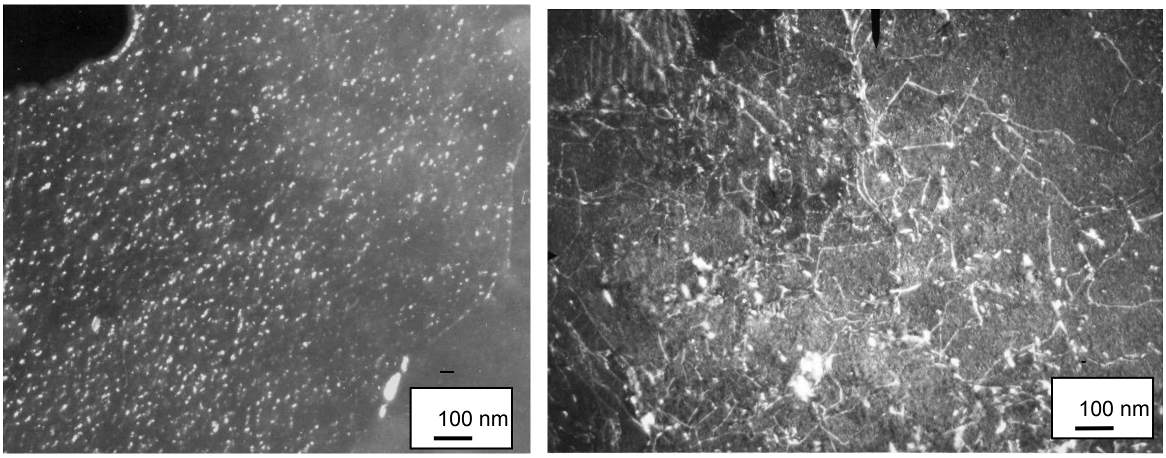
1. B.Gurovich et al., J. Nucl. Mat., v.264 (1999) p.333.
2. B.Gurovich et al., J. Nucl. Mat., in progress (2009).



a b

Figure 1. SEM micrograph: a - brittle intergranular fracture surface, b – ductile intergranular fracture surface.



a b

Figure 2. TEM micrographs (dark-field images) of the fine structure of pressure vessel steel: a- nanosize precipitates, b - radiation defects – dislocation loops.

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