Role of lithium in the formation of He bubbles in boron-alloyed steel after neutron irradiation

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Formation of He bubbles in martensitic steels during neutron irradiation is the most important factor influencing their mechanical properties. The formation of overpressed He bubbles leads to material swelling, followed by the loss of ductility and an increase in hardness and embrittlement. Since decades, the alloys containing natural boron or ^{10}B have been used as models to study the He influence on material properties [1]. Helium inside alloys is generated by the nuclear reaction $^{10}B(n, \alpha)^{7}Li$ after doses significantly lower than the He concentration reached by neutron irradiation only. All previous studies considered He the only factor influencing bubble formation. On the other hand, Li, which is insoluble in iron, will be produced in the same concentration as He and is also supposed to have an influence on the formation of He bubbles.

The European reduced-activation ferritic-martensitic (RAFM) reference material EUROFER 97 was alloyed with ^{10}B to study the influence of He on the mechanical properties of irradiated material [1]. It was shown by investigations of unirradiated specimens that the boron inside alloys partly forms BN precipitates of 200-500nm size. In the area around these precipitates the concentration of He and Li increases significantly compared to the specimen average. Previous investigations clearly revealed the formation of two damaging halos due to recoiling lithium ions and alpha particles. Calculations of Li and He recoil ranges are in good agreement with the respective radii of the halos observed in the electron microscope (Fig. 1).

TEM investigations of the specimen alloyed with 1160 wt. ppm ^{10}B irradiated at 300°C show that the halo can be divided into three parts (Fig. 1b). The first part has a radius down to 1.1µm measured from the middle of the particle (hole after irradiation). The second part of the halo is located between 1.1µm and 1.5µm measured from the centre and is characterised by a high density of He bubbles with diameters ranging from 2-3nm to 25 nm. The third halo is located in the range from 1.5µm to 2.6µm. Similar to the second halo, bubbles with a smaller diameter (3nm - 8nm) and a larger diameter (10nm - 15 nm) can be observed.

It is known from the phase diagram that Li is insoluble in iron and subsequently Li should precipitate during irradiation. Detection of Li inside the Fe/Cr alloy using EELS is a challenging task. The Li K EELS edge (58eV) is characterised by a smooth shape and overlaps with Fe-$M_{2,3}$ (54eV) and Cr-$M_{2,3}$ (51eV). The clear separat It was found to be helpful that the plasmon peak of metallic Li shows a pronounced feature at 13 eV [2] (Fig. 2). Using this feature, detection of Li and its clear separation from other matrix elements and even from He are possible (Fig. 2d). In the Fig. 2a-d are presented plasmon spectra obtained from the different areas, the Fe/Cr matrix, He bubble, Li drop and area Li with He. The contribution from Li and He could be clearly separated the plasmon peak from the matrix (Fig. 2b, c). Application of spectroscopic EELS mapping allows for imaging Li drops inside large He filled bubbles (Fig. 3). Li drops were detected only in the bubbles located in the area of the second halo (Fig. 1) on the inner wall.

The irradiation at high temperature leads to the diffusion of Li atoms and formation of bubbles which are filled with He as well as with Li. The largest bubbles were observed in the second halo which correlates with Li distribution. The presented results allow conclusions that Li promote the formation of large He filled bubbles in the second halo.

References

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[2] C. Ahn, „ Transmission Electron Energy Loss Spectrometry in Materials Science and the “Wiley-VCH Verlag second edition (2004)

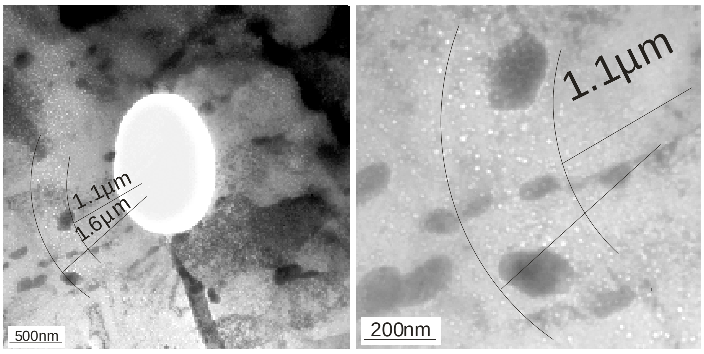

Figure 1 *The hole inside the matrix shows the location of the BN particle before irradiation. Around the particle the formation of pronounced damaging halo was detected.*

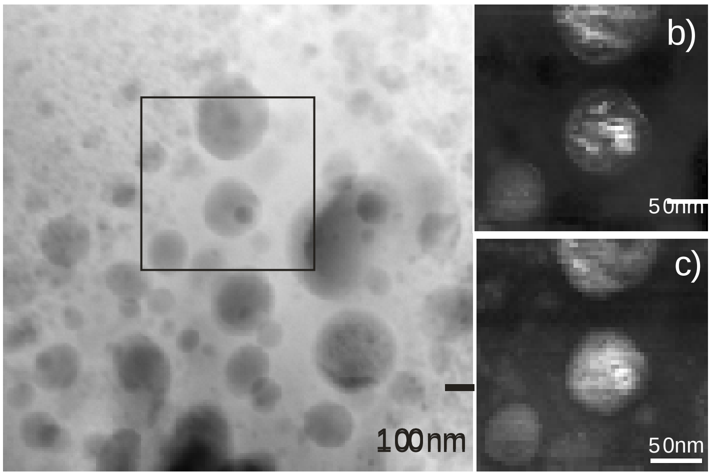

Figure 3 *HAADF image of the area with bubbles (a) and 2 dimensional maps (b) and (c) showing spatial distribution of Li and He respectively.*

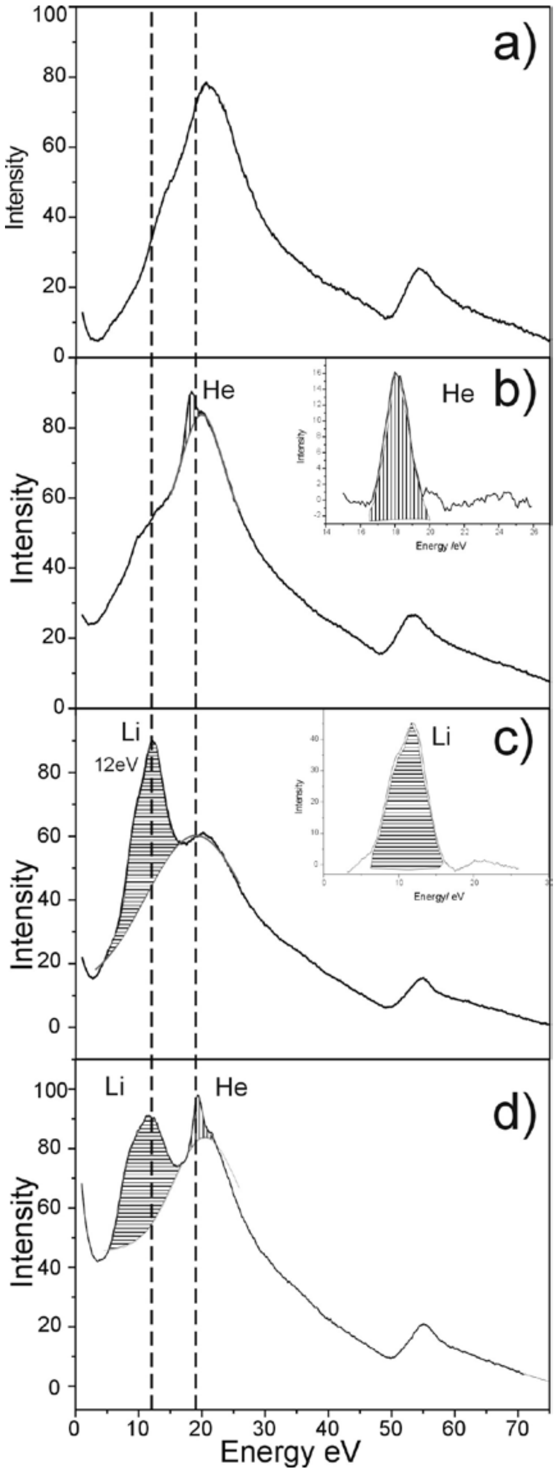

Figure 2*. Low-energy EELS spectra: (a) Fe/Cr matrix, (b) He-filled bubble, (c) bubble with Li, (d) bubble with He and Li.*