Evolution of radiation induced dislocation loop in low carbon Mn-Ni-Mo steel

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1. Introduction

Low carbon Mn-Ni-Mo bainitic steel has been used for a nuclear reactor vessel in PWR. Recently it is going to be adopted to a candidate material for advanced reactor vessel in VHTR [1]. Although it has been known that it is well optimized and reliable structural material used for commercial nuclear reactor vessel, it is necessary to improve performance of resistance to irradiation induced degradation to be used in VHTR. The irradiation induced degradation behavior of structural material depends on alloying element, fabrication processing and the resulting microstructures [2,3]. It has been understood that they affect the evolution of microstructural defects such as dislocation loop and void which are important products to give rise to radiation hardening and swelling.

In this work, we observed microstructure change with heat treatment processing in low carbon Mn-Ni-Mo steel using TEM and investigated the relation between resultant microstructure and microstructural defects formed by ion irradiation.

2. Experimental

The chemical composition of a low carbon Mn-Ni-Mo steel for the present work was: Fe-0.19C-1.35Mn-0.82Ni-0.51Mo-0.17Cr-0.009Al-0.008Si (wt-%). The experimental samples with 3mm thickness were prepared using micro cutter. And they were water quenched after austenizing heat treatment at 1173K during one hour and then were tempered at 923K with different holding time. Figure 1 shows schematic diagram about heat treatment conducted in this work. They were polished mechanically for the thickness reduction and thinned electro-chemically for removal of any damage formed on their surface.

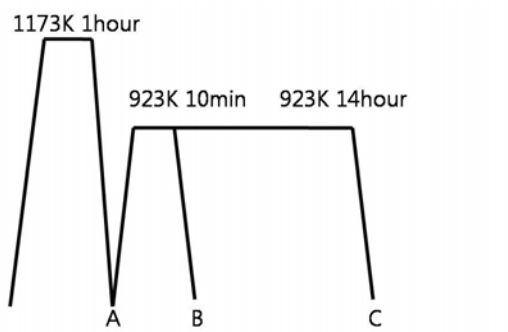

Figure 1 Schematic diagram about heat treatment conducted in this work

Atomic displacement damage was introduced by 8MeV Fe^{4+} ions accelerated with an ion accelerator in the Korea Institute of Geoscience & Mineral Resources (KIGAM). The experimental samples were irradiated at the fluence in the order of 5.8×10^{14} ion/cm^2 (1dpa at peak damage depth). Irradiation temperature is about 673K~773K. Thin foils for TEM observation were prepared with a focused ion beam (FIB) micro-processing device and low energy ion miller. A TEM specimen was fabricated using FIB micro-processing device to obtain TEM specimen in a specific region. TEM observation was performed by using a 200keV JEOL 2000FXII and JEOL 2100F microscope. Weak beam imaging by means of TEM has been conducted for the observation of dislocation, dislocation loop and void.

3. Results and discussion

3.1 Microstructure in experimental samples

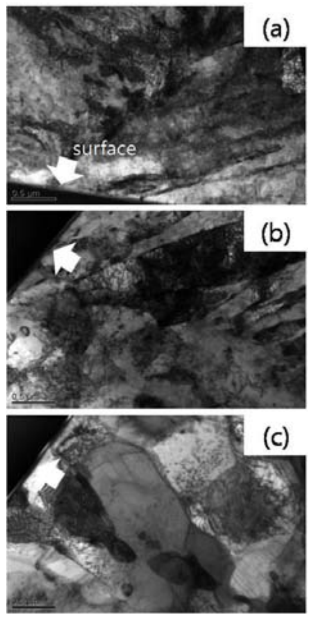

Figure 2 TEM image with low magnification of sample A (a), sample B (b) and sample C(c)

Figure 2(a) shows TEM micrographs of the untempered sample A after irradiation experiment. Briefly, it is found that the untempered sample A has martensite microstructure composed of narrow and parallel ferrite laths. In the lath, many dislocations are tangled, which represented dark lines or dark clouds in TEM image. It is thought that the dislocation structure is generated during air cooling process. No irradiation induced defects and carbides are found in untempered sample A. Figure 2(b) presents the TEM micrograph of the sample B tempered during 10 min. Dislocation density in sample B is lower than that in sample A and small carbides are nucleated on lath boundary and grain boundary. Figure 2(c) shows the TEM micrograph of the sample C tempered during 14 hour. The dislocation density in sample C is much lower than that in sample A and sample B. Also the size of lath is increased and the lath boundary is not straight line compared with other samples. Long tempering time gives rise to the growth of lath and amalgamation between laths. Micrometer sized carbides formed during tempering treatment are observed in the lath boundary. It is considered as a cementite (Fe_3C) which is a stable phase in the tempering temperature.

3.2 Microstructural defects in experimental samples

In case of sample B tempered during 10 min, microstructural defect is not observed easily below about 1um to the surface. On the other hand, in the case of sample C tempered during 14 hour, dislocation loops are gathered below about 1 and 2 um from the surface. For the detail observation of the existence of dislocation loops which might be formed by ion irradiation, we conducted the weak beam imaging at g=110 and 002. Figure 3 shows TEM images below 1um to the surface of samples tempered during 10 min and 14 hour. Both the irradiated samples contained dislocation loops between 1μm and 2μm from a surface. They were considered as dislocation loops with a Burgers vector (**b**) formed in the Fe alloy by an irradiation with a_0<100> and 1/2 a_0<111> components [4]. The average size and number density of the dislocation loops are larger in the sample C when compared with that in the sample B. As we have seen early, dislocation density of the sample B is much higher than that of sample C. It seems reasonable to suppose that the existence of dislocation is a stronger sink site for annihilation of defect clusters, which leads to a considerable suppression to the formation and growth of dislocation loop.

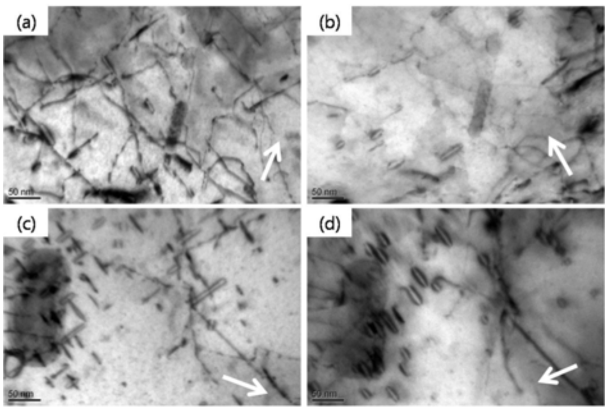

Figure 3 Dislocation loop formed by ion irradiation in sample B (a,b) and sample C (c,d)

4. Conclusions

TEM observation was performed to investigate the microstructural defect evolution in low carbon Mn-Ni-Mo steel with tempering holding time. Under a Fe ion irradiation, Dislocation loops were observed in samples tempered during 10 min and during 14 hours, on the other hand, they were not in untempered sample. As holding time is increased, the growth and formation of dislocation loop is enhanced due to reduction of density of dislocation which is sink site to annihilate defect clusters.

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