

Nanostructures Formation in Al-B₄C Neutron Absorbing Materials after Accelerated Irradiation and Corrosion Tests

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Al/B₄C metal matrix composite (MMC) is an important neutron absorbing material used in both wet storage pools and dry storage casks of spent nuclear fuel for preventing criticality. Because of the high neutron absorption cross-section of ¹⁰B, the material can effectively absorb fast and thermal neutrons, but at the same time suffers neutron irradiation damage from the nuclear spent fuel. Moreover, interactions of fast and thermal neutrons with boron lead to the production of several transmutation species such as helium, lithium and others, according to the transmutations reactions induced by neutron irradiation, including the ¹⁰B (n, ⁴He) ⁷Li reaction and others. The most abundant product of these reactions is energetic helium (~1.47MeV), which may induce further radiation damage and be precipitated out in the material as helium bubbles. The formation and coalescence of the helium bubbles in boron carbide and on boundaries, especially phase boundaries, would affect the corrosion performance of the material in its working environment. The degradation of the material may lead to boron loss which is the critical element to control the criticality of spent fuel. To evaluate the radiation and corrosion effects for interim storage of two hundred years accelerated He⁺ irradiation and corrosion tests were carried out.

The bulk MMC sample was irradiated with 400keV He⁺ to 1.5×10¹⁷ ions/cm² and 50keV He⁺ to 3.5×10¹⁶ ions/cm² at room temperature. Autoclave corrosion tests of the ion-irradiated samples were carried out at 400°C for 112 days, and humidification of the argon was accomplished by passing the argon through water. Microstructures of irradiated and corroded material were characterized by SEM, TEM and STEM. TEM samples were prepared by Focus Ion Beam (FIB) lift-out method. TEM analysis and characterization were conducted with a JEOL HRTEM 3011 and a JEOL 2100F STEM and JEOL 3100R05 TEM/STEM at the Electron Microbeam Analysis Laboratory (EMAL), University of Michigan.

SEM images in Figure 1 show the evidence of corrosion. It is apparent that the boron carbide particles on the surface were totally corroded and disappeared after the corrosion test. Fiber-like nanostructures formed at the bottom of the hole, where was the interface between Al matrix and B₄C particles. TEM investigation was taken on the cross-section sample in order to study the newly formed nanostructures. Figure 2a indicates that the nanostructures formed at both surface and the interior. The interior nanostructures are along the Al/B₄C phase boundaries. After He⁺ irradiation, helium bubbles formed and coalesced at the phase boundaries as noted in Figure 2b [1]. These helium bubbles may lead to formation of open channels causing the reaction of interior material with water. Analysis of EDS point spectra shows that there is a newly formed phase at the interface [2] after the sample was placed at 400°C for 112 days. The SAD pattern and high resolution BF STEM images revealed the crystalline nature of the nanofibers.

Reference

[1] F. Zhang, et al, *Microsc. Microanal.* 19 (Suppl 2), 2013.

[2] Z. Zhang, et al, *Metallurgical and Materials Transactions A*, 43A (2012) 281.

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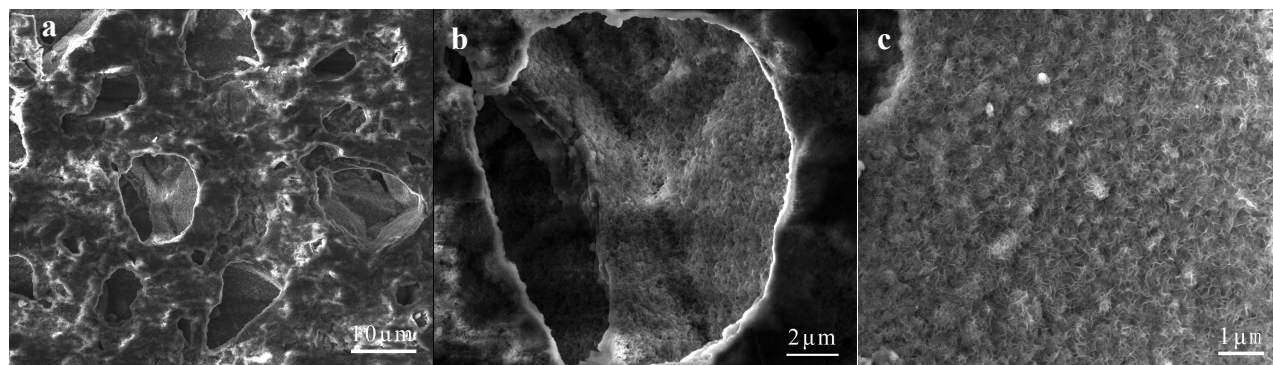


Figure 1. (a) SEM image of the sample surface after ion irradiation and corrosion test; (b) SEM image of a hole where a boron carbide particle was dissolved; (c) SEM image at high magnification showing the nanostructures at bottom of the hole.

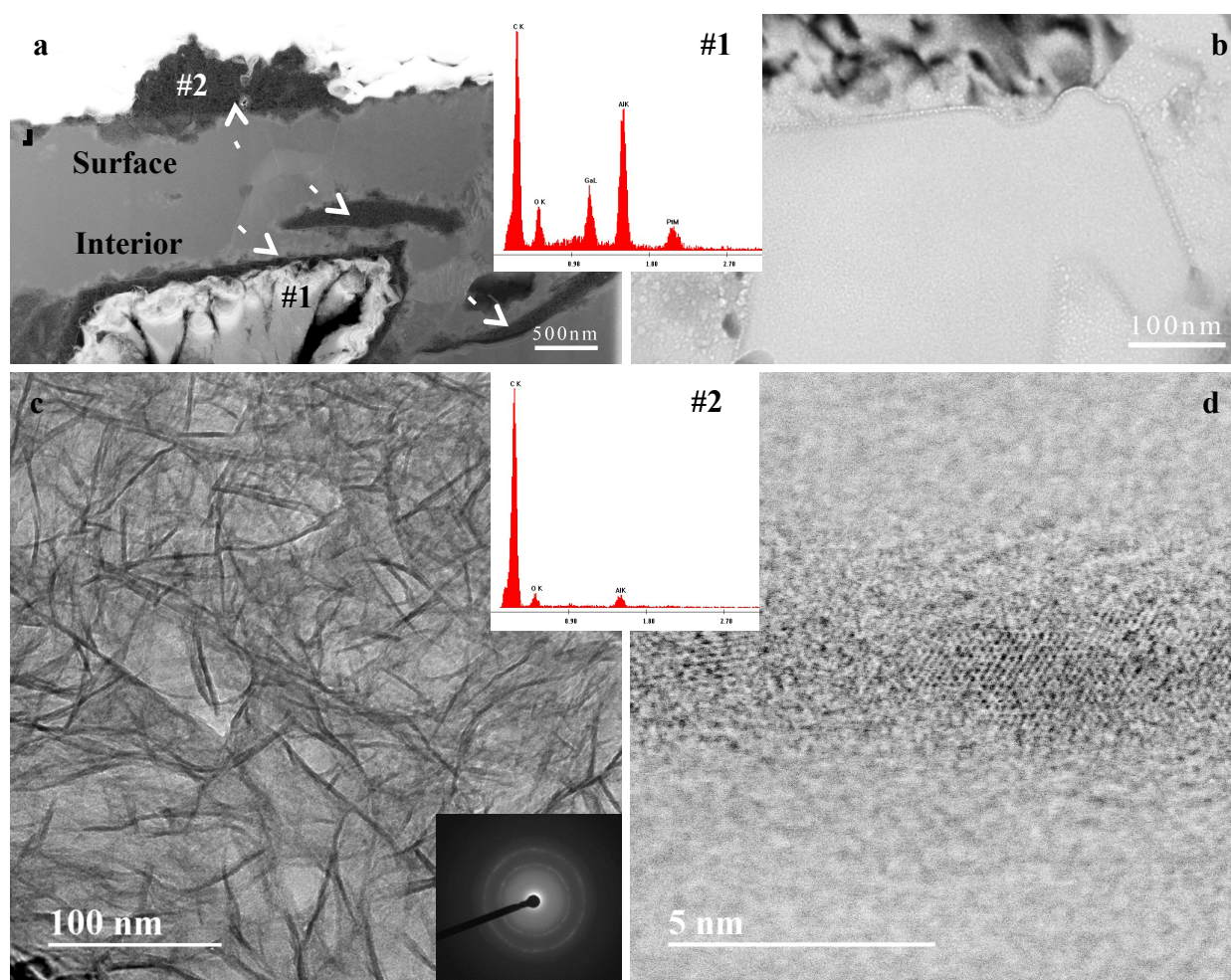


Figure 2. (a) HAADF image and (b) BF STEM image of the near surface area of a corroded sample, the nanostructures were marked by arrow, (c) TEM BF image of “#2” area at high magnification and corresponding SAD pattern, (d) high resolution BF STEM image of the nanocrystal. The insets are EDS result of point #1 (area near nanostructures) and #2 (on the nanostructure).