Multi-step Internal Nitriding of Tungsten-titanium Alloys

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Abstract

Internally nitrided dilute W-Ti alloy specimens having a heavily deformed surface microstructure were prepared by a multi-step internal nitriding at 1573-2073 K. Primary nitriding below their recrystallization temperature induced a precipitation of ultrafine TiN particles. After secondary and tertiary nitriding, those precipitates grew into rod-like TiN with a length of 20-60 nm. The recrystallization temperature after nitriding was elevated above 2073 K. The yield strength at 1773 K obtained from nitrided W-0.5 mass% Ti alloy was about 5 times as large as that of the recrystallized specimen. DBTT of the nitrided alloys was about 373 K.

Keywords : tungsten alloy, internal nitriding, recrystallization temperature, ductile-to-brittle transition temperature

1. Introduction

Tungsten alloys are promising candidates for high-temperature structural materials, such as plasma facing components of fusion reactors. However, their applications have been limited by recrystallization embrittlement. Recrystallization leads to grain coarsening, resulting in a significant increase in the ductile-to-brittle transition temperature (DBTT). Thus, it is necessary to suppress recrystallization, i.e., raise the recrystallization temperature, for effective use of tungsten alloys.

Our recent studies on internal nitriding of dilute Mo-Ti alloys[1,2)] have shown that the multi-step internal nitriding[3)] is effective in raising the recrystallization temperature of those alloys. Using this technique, it is possible to disperse fine TiN particles in those alloys, with the deformed microstructure retained owing to the gradual, step-by-step increase in nitriding temperature from below the recrystallization temperature. Since these TiN particles act effectively as pinning point for grain boundary migration, the recrystallization temperature of Mo-Ti alloys is elevated above 1873 K.

In this study, we subjected dilute W-Ti alloys to a multi-step internal nitriding in an attempt to improve their recrystallization temperature and mechanical properties.

2. Experimental

W alloys containing 0.5-1.5 wt% Ti (hereafter "W-*x*Ti" where x denotes wt%) were prepared by a powder metallurgy method. Mechanical and electrolytic polishing were performed on rectangular specimens ($w2.0 \times t1.0 \times l20$ mm) cut out from as-rolled sheets. Nitriding was performed at 1573 to 1973 K in flowing N_2 at 0.1 MPa. The specimens were first subjected to primary nitriding at 1573 K for 49 h. Then, secondary and tertiary nitriding were performed at 1773 and 1973 K for 25 h. For comparison, recrystallized specimens were prepared by heating as-rolled alloys at 1973 K in a vacuum of about 1.4×10^{-4} Pa.

The cross-section of the specimens was examined by optical microscopy. The deformed microstructure and precipitates were studied by transmission microscopy (TEM). Micro-Vickers hardness was measured by using a weight of 25 gf for 15 s at room temperature. In order to examine DBTT, three-point bending tests were performed at temperatures from 293 to 423 K at a crosshead speed of 0.39 mm/min. The crack initiation area was investigated by fractographic analysis using a scanning electron microscope (SEM). High temperature three-point bending tests were performed at 1773 K in a vacuum of about 5.0×10^{-4} Pa at a crosshead speed of 0.2 mm/min.

3. Results and discussion

Fig. 1 shows an optical micrograph of the cross-section of W-0.5Ti alloy subjected to three-step nitriding. Although recrystallization was observed in the central region, the specimen surface retained a deformed microstructure. The thickness of the region where the recrystallization was suppressed depended on the primary nitriding time.

Fig. 2 shows a TEM image taken near the specimen surface of W-0.5Ti alloy subjected to three-step nitriding. Rod-like precipitates with a length of 20-60 nm are clearly visible. Grain boundaries of the tungsten matrix were pinned by these precipitates, as indicated by the white arrows in the figure. Electron diffraction analysis showed that the precipitates were TiN with an fcc structure. Thus, a dispersion of TiN precipitates can be formed by the multi-step internal nitriding without recrystallization of the tungsten matrix.

Fig. 3 shows recrystallization temperatures of specimens before and after three-step nitriding. It is found that recrystallization temperatures after nitriding were elevated above 2073 K. This is about 400 degrees higher than recrystallization temperatures of the as-rolled specimens.

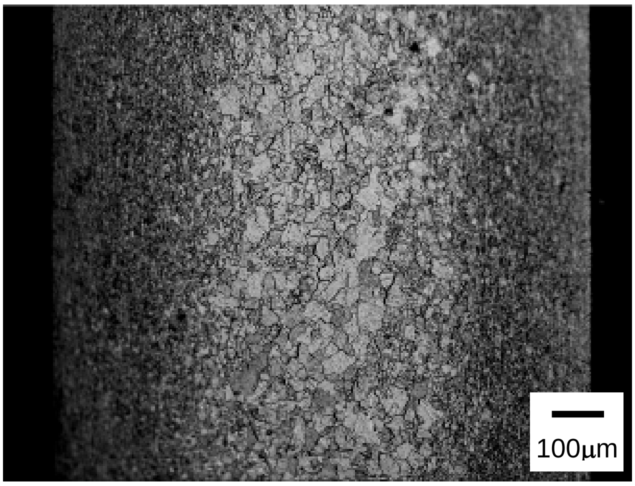

Fig. 1. Optical micrograph of the cross-section of W-0.5Ti alloy subjected to three-step nitriding.

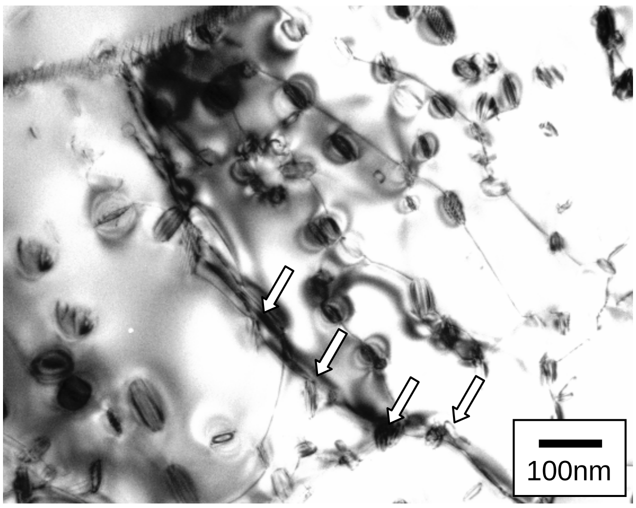

Fig. 2. TEM image taken near the specimen surface of W-0.5Ti alloy subjected to three-step nitri ding.

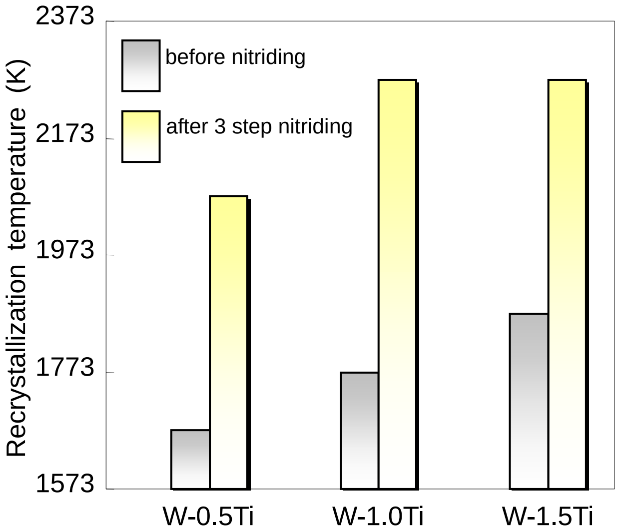

Fig. 3. Recrystallization temperatures of specimens b efore and after three-step nitriding.

Mechanical properties after the three-step nitriding were evaluated by three-point bending tests. Regardless of the titanium content of the alloys, DBTTs of the nitrided specimens were about 373 K. This is about 50 degrees lower than DBTTs of the recrystallized specimens. The yield strength at 1773 K obtained from nitrided W-0.5Ti alloy was about 5 times as large as that of the recrystallized specimen.

4. Summary

The multi-step internal nitriding behavior of dilute W-Ti alloys and mechanical properties of the resultant nitrided specimens were studied. We can conclude that multi-step internal nitriding is highly effective in the suppression of recrystallization embrittlement and strengthening of tungsten alloys containing nitride-forming elements like titanium.

5. References

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