# Preparation of TZM Alloys Having Elongated Coarse-grain Structure with High Aspect Ratio and their Mechanical Properties

Gouhei Toyosima  $^{1,a}$ , Masahiro Nagae  $^{1,b}$ , Tetsuo Yoshio  $^{1,c}$ , Jun Takada  $^{2,d}$ , Yutaka Hiraoka  $^{3,e}$ , Tomohiro Takida  $^4$ 

<sup>1</sup>Graduate School of Environmental Science, Okayama University, 3-1-1, Tushima-naka Okayama 700-8530, Japan, <sup>2</sup>Graduate School of Natural Science and Technology, Okayama University, Okayama Tushima-naka 700-8530, Japan, <sup>3</sup>Department of Applied Physics, Okayama University of Science, Okayama 700-0005, Japan, <sup>4</sup>A.L.M.T. TECH Inc, 2 Iwase-koshi-cho, Toyama 931-5843, Japan <sup>a</sup>gev16141@cc.okayama-u.ac.jp, <sup>b</sup>nagae@cc.okayama-u.ac.jp, <sup>c</sup>yoshio@cc.okayama-u.ac.jp, <sup>d</sup>jtakada @cc.okayama-u.ac.jp, <sup>e</sup>hiraoka@dap.ous.ac.jp

#### Abstract

TZM alloy having elongated coarse-grain structure was developed by three-step internal nitriding treatment at 1423 to 1873 K in  $N_2$  and subsequent recrystallization treatment at 2173 K in vacuum. Some specimens were subjected to re-nitriding treatment at 1873 K for 16 K. After the recrystallization treatment, aspect ratio (K) of grains for rolling direction was about 50 at the maximum. Yield stress obtained at 1773 K after re-nitriding treatment was about 6 times as large as that of recrystallized specimen. Re-nitriding was very effective in the improvement in strength of TZM alloy having elongated coarse-grain structure.

Keywords: molybdenum, TZM, multi-step internal nitriding, elongated coarse-grain structure

#### 1. Introduction

Molybdenum and its alloy are promising candidates for high-temperature structural materials such as plasma facing components and so on. However, the effective application development and the practical use of these metals have not been performed enough because of its high intergranular failure susceptibility by the recrystallization. It is therefore, necessary to develop molybdenum alloys having high resistance to recrystallization embrittlement. Molybdenum alloy which is prepared by adding a proper amount of rare earth oxides(La<sub>2</sub>O<sub>3</sub>, Y<sub>2</sub>O<sub>3</sub> etc.) and by adding a large degree of deformation is called "doped molybdenum". Typical microstructure of this material after recrystalization is an elongated coarse-grain structure<sup>1</sup>. Doped molybdenum having the elongated coarse-grain structure shows superior creep resistance at high temperatures than pure molybdenum having an equiaxed small-grain structure.

Our recent study on multi-step internal nitriding of TZM alloy (Mo-0.5%Ti-0.08%Zr-0.03%C) has shown that abnormal grain growth occurs after vacuum annealing for the nitrided TZM alloy<sup>2</sup>. The aim of this study is to prepare a TZM alloy having an elongated coarse-grain structure and to evaluate its mechanical properties. In addition, we attempted strengthening of coarse-grained TZM alloy by re-nitriding.

# 2. Experimental

Commercial TZM alloy (Mo-0.5% Ti-0.08% Zr-0.03% C)

sheet with thickness of 0.5mm was used as the starting material. Rectangular specimens with dimensions of  $2.0^w \times 20^l \times 0.5^t$  mm were cut out from the sheet in parallel to rolling direction, and mechanical and electrolytic polishing were carried out.

Three-step internal nitriding was performed at 1423 K for 25 h, 1573 K for 16 h, and 1873 K for 9 h in a flowing N<sub>2</sub> gas, and subsequent recrystallization treatment was performed at 2173 K in vacuum. Some specimens were subjected to re-nitriding treatment at 1873 K for 16 h. For comparison, La<sub>2</sub>O<sub>3</sub>-doped Mo alloys were recrystallized at 2173 K in vacuum (here after "doped Mo"). Grain growth behavior was investigated by optical and transmission electron microscopy (TEM) and mechanical properties were examined by Vickers hardness measurements and static three-point bend tests.

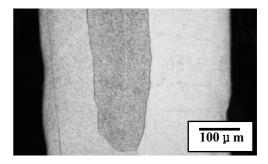


Fig. 1. Optical micrograph of cross section for TZM heated for 1 h at 2173 K after multi-step internal nitriding.

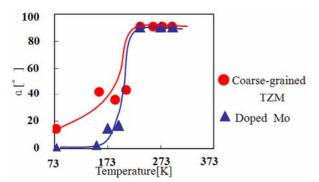


Fig. 2. Relationship between bend angle and test temperature for coarse-grained TZM alloy and doped Mo.

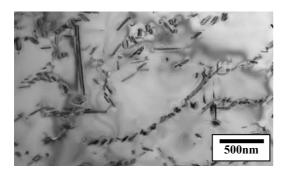


Fig. 3. TEM image of coarse-grained TZM alloy after re-nitriding at 1873 K for 1 h.

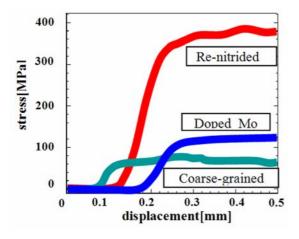


Fig. 4. Stress-displacement curves obtained at 1773 K for specimens before and after re-nitriding and doped Mo.

## 3. Results and Discussion

Figure 1 shows optical micrograph for cross section of specimen heated in a vacuum for 1 h at 2173 K after three-step internal nitriding. It is found that the whole of specimen changed to elongated coarse-grain structure. After the recrystallization treatment, aspect ratio (L/W) of grains for rolling direction was about 50 at the maximum.

Figure 2 shows the relationship between bend angle and test temperature for coarse-grained TZM alloy. The data for doped Mo specimens were also shown for comparison. The DBTT (ductile-brittle transmission temperature) of coarse-grained TZM alloy was lower than that of doped Mo (DBTT≒153 K). It is notable that coarse-grained TZM exhibits ductility even at liquid nitrogen temperature. Thus, coarse-grained TZM is superior in low-temperature ductility. However, hardness of coarse-grained TZM was almost equal to the recrystallized TZM alloy having equiaxed grain structure. TEM observation revealed the complete decomposition of the TiN precipitates during recrystallization treatment at 2173 K for 1 h.

These results suggest that precipitation hardening is possible by re-nitriding of coarse-grained TZM. Fig.3 shows typical TEM image near the specimen surface after re-nitriding at 1873 K for 16 h. Fine TiN precipitates were clearly observed. After re-nitriding, hardness increased by about 100 Hv due to precipitation hardening.

Figure 4 shows stress-displacement curves obtained at 1773 K for specimens before and after re-nitriding. The data for a doped Mo are also shown for comparison. Yield strength after re-nitriding was about 3 times as large as that of doped Mo and about 6 times as large as that before the re-nitriding.

### 4. Summary

- •TZM alloy having elongated coarse-grain structure was successfully developed by three-step internal nitriding treatment at 1423 K to 1873 K in N<sub>2</sub> gas and subsequent recrystallization treatment at 2173 K in vacuum.
- •DBTT of the coarse-grained TZM was lower than 77K.
- •Re-nitriding was very effective in the improvement in strength of TZM alloy having elongated coarse-grain structure.

#### 5. References

- 1. T. Igarashi, Materia Japan, 41[5], p.366(2002).
- 2. M. Nagae, T. Yoshio, J. Takada, and Y. Hiraoka, Materia ls Transactions, **46**[10], p.2129(2005).