Strength Properties of Yttrium-Oxide-Dispersed Tungsten Alloy*

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The microstructure of newly developed yttrium oxide dispersed tungsten alloy was investigated by examining the effect of sintering temperature on the particle size of yttrium oxide and the crystal size of tungsten. Also, it was confirmed that the bending strength of yttrium oxide dispersed tungsten alloy was more strongly affected by the sintering temperature in comparison with a sintered tungsten sample. The residual stress, induced by the coefficient of thermal expansion mismatch, is analyzed for the tungsten matrix composite with a particle of yttrium oxide using the finite element method. Because of the high residual stress, particles of yttrium oxide become crack initiation sites under the fabrication process. Finally, it is also shown that the bending strength of yttrium oxide dispersed tungsten alloy can be estimated simply by the fracture mechanics approach, based on the assumption of a flaw introduction effect by the yttrium oxide dispersion.

Key Words: Bending Strength, Composite Material, Residual Stress, Fracture Mechanics, Sintering, Yttrium Oxide-Dispersed Tungsten Alloy, Pure Tungsten

1. Introduction

Tungsten exhibits a high high melting point (3653 K), a small thermal expansion and a low vaporization pressure in comparison with other metals and is widely used in electronic and high-temperature vacuum equipment. Also, tungsten is useful as a crucible material for melting of reactive materials such as rare earth metals. However, the grain boundary of tungsten is often selectively attacked by molten metals. It has already been confirmed by the authors that yttrium oxide dispersed in the tungsten results in excellent corrosion resistance against the molten metals1,2. Also, the ceramic particles dispersed alloy shows high strength at elevated temperatures due to the strengthening effect of ceramic particles3 and the ductility is superior to that of ceramics because the almost all matrix of the alloy is metal.

Thus, the ceramic-particles-dispersed alloy is expected to combine the superior mechanical properties of both ceramics and metals. For that reason, it is important to optimize the size and the additive volume of ceramic particles. In case of the Y2O3-dispersed W alloy, it is desirable to increase the addition of Y2O3 to improve the corrosion resistance, but at the same time, the strength and ductility tends to decrease with increasing the additive volume of micron-order Y2O3 particles.

In this study, the bending strength of Y2O3-dispersed W alloy is investigated by experimentally examining the effect of additive volume of Y2O3 particles and sintering temperature on the microstructure. Furthermore, the fracture mechanism of Y2O3-dispersed W alloy is investigated and makes possible quantitative evaluation of the static strength of Y2O3 dispersed W alloy using the method of fracture mechanics.

2. Materials Used and Experimental Details

In this study we used tungsten powder (W: 2.09 μm particle diameter and 99.9% purity) and yttrium powder (Y2O3: 1.36μm particle diameter and 99.9% purity). Tungsten powders with 5 vol%, 10 vol% and
20 vol% $Y_2O_3$ particles were mixed and crushed using a pottery ball mill for 604.8 ks and were deoxidized at 873 K for 3.6 ks in hydrogen atmosphere. The powders were compacted to 65 mm diameter and 30 mm thickness by cold isostatic pressing (CIP) at 196 MPa. After pre sintering at 1873 K for 28.8 ks in vacuum ($<6.7 \times 10^{-4}$Pa), the sintering was carried out at one of three temperatures, 2073 K, 2273 K or 2473 K for 28.8 ks in vacuum ($<6.7 \times 10^{-4}$Pa). Specimens were cut out of the sintered block and machined. The pure tungsten specimens were also fabricated in the same way, namely the pure tungsten powder was crushed by ball-milling and was sintered in vacuum.

The density of sintered specimens was measured by the Archimedean method. The relative density was calculated using the real density such as W: 19.25 g/cm$^3$ and $Y_2O_3$: 5.03 g/cm$^3$. Four point bending specimens (4 mm in width, 3 mm in thickness and 40 mm in length) were tested at room temperature (296 K) and 0.5 mm/min as the displacement speed, according to Japanese Industrial Standards (JIS R 1601).

3. Experimental Results

3.1 Microstructure and sintering density

Figure 1 shows typical examples of the microstructure of the $Y_2O_3$ dispersed W alloy in comparison with the pure sintered tungsten. It can be observed that the $Y_2O_3$ particles of size 2-3 μm exist in the boundary of W crystals. Meanwhile, pores can be observed in the boundary of W crystals in the case of the pure sintered W. Also, the crystal size of W is markedly affected by the additive volume of $Y_2O_3$ particles.

The effect of sintering temperature on the average crystal size of W is shown in Fig. 2. In both $Y_2O_3$ dispersed W alloy and pure sintered W, the average crystal size of W grows larger with increasing sintering temperature. And the growth of W crystals is suppressed with increasing additive volume of $Y_2O_3$ particles. However, as shown in Fig. 1 (sintering temperature: 2473 K), the average crystal size of W in the $Y_2O_3$-dispersed W alloy grows larger than that of the pure sintered W in case of the elevated-temperature sintering. This is because the sintering of $Y_2O_3$ proceeds rapidly at the elevated temperature and the sintering of W is improved by the added $Y_2O_3$ particles. Figure 3 shows the effect of sintering temperature on the size of $Y_2O_3$ particles. The size of $Y_2O_3$ particles grows larger with increasing sintering temperature and this tendency is marked with increasing the additive volume of $Y_2O_3$ particles. The measured pore sizes of pure sintered W are shown in Fig. 3. This is the reason that the sintering proceeds at a high rate with increasing the sintering temperature and the pore size becomes smaller.

Figure 4 shows the effect of sintering temperature on the relative density of the $Y_2O_3$ dispersed W alloy. The relative density increases with increasing sintering temperature. This tendency becomes more marked by increasing the additive volume of $Y_2O_3$ particles. Namely, the relative density of pure sintered W is 85% in the case of 2273 K sintering, and relative density as high as 97% can be obtained in the
Fig. 3 Effect sintering temperature on particle size of Y$_2$O$_3$

![Diagram showing the effect of sintering temperature on particle size of Y$_2$O$_3$.]

Fig. 4 Effect of sintering temperature on relative density case of the Y$_2$O$_3$-dispersed W alloy. Relative density over 99% is obtained at 2073 K sintering in the case of the 20 vol.% Y$_2$O$_3$ dispersed W alloy. Adding the Y$_2$O$_3$ particles is useful for reducing the sintering temperature. Also, it is estimated that the strength of Y$_2$O$_3$-dispersed W alloy is affected by the sintering temperature and the additive volume of Y$_2$O$_3$ particles.

3.2 Bending strength properties

The densification of Y$_2$O$_3$-dispersed W alloy is useful for improving the corrosion resistance that is the most important property of the W alloy. On the other hand, this densification has an important effect upon the strength.

Typical examples of four point bending strength of the pure sintered W and the 20 vol.% Y$_2$O$_3$-dispersed W alloy are shown in Figs. 5(a) and (b), respectively. Eight specimens were used to test bending strength at room temperature. Each specimen was fractured under the same conditions, i.e., the load vs. deflection curves were straight lines. Thus, the fracture stress, that is four point bending strength, was calculated from the highest load point. Figure 5 shows the two parameter Weibull distributions of the four point bending strength. The relationships between the accumulated probability using the mean rank method and the four point bending strength are well represented by a straight line for each material and each sintering temperature. The shape parameter, $m$, and the mean value, $\mu$, are indicated in each figure. It may be assumed that the shape parameter.
Table 1  Material constants used for FEM analysis

<table>
<thead>
<tr>
<th></th>
<th>Young's modulus E (GPa)</th>
<th>Poisson's ratio ν</th>
<th>Thermal expansion coefficient at 2273K ( a (10^{-6}/K) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>277</td>
<td>0.28</td>
<td>5.4</td>
</tr>
<tr>
<td>Y₂O₃</td>
<td>175</td>
<td>0.28</td>
<td>10.6</td>
</tr>
</tbody>
</table>

Fig. 6  Effect of sintering temperature on bending strength

\( m \), is not strongly dependent on materials or the sintering temperature used in this experiment. However, the mean value, \( \mu \) of bending strength increases with increasing sintering temperature. In contrast, the bending strength of the 20 vol\% Y₂O₃ dispersed W alloy decreases with increasing sintering temperature. The details of these strength characteristics are shown in Fig. 6. As described above, the bending strength of the Y₂O₃ dispersed W alloy decreases with increasing sintering temperature. This tendency is marked for W alloy with many Y₂O₃ particles. The pure sintered W shows the reverse tendency in that the bending strength increases with increasing the sintering temperature. It seems that the formation of pores in the pure sintered W can be prevented by elevated temperature sintering and the bending strength can be increased. In the case of the Y₂O₃ dispersed W alloy, the relative density is improved with increasing additive volume of Y₂O₃ particles, and the strength reduction is not due to the presence of pores. It is thus considered that the large Y₂O₃ particles have an important effect upon the strength of W alloy.

4. Consideration for Strength Evaluation

The authors have previously investigated the residual stress characteristics of ceramic coatings and clarified that the residual stress, induced by coefficient of thermal expansion mismatch, affects the cracking of coating films. Similarly, some analytical investigations have been carried out to determine the stress distribution of the particle dispersion strengthened composite around the dispersed particles. In particular, it is considered to be very important to clarify the residual stress in the vicinity of Y₂O₃ particles during the fabrication process for investigating the strength behavior of Y₂O₃ dispersed W alloy.

From the point of view described above, the thermoelastic finite element method (the rectangular elements in the plane strain state) was used to determine the thermal stresses generated when the Y₂O₃ dispersed W alloy was heated uniformly to the fabrication temperature and then cooled down to room temperature (difference in temperature, \( \Delta T \)). The thermal stress obtained in this analysis is opposite in sign but equal in absolute value to the thermal stress generated during the uniform heating for the sintering. The analysis assumed that analysis models were perfect elastic bodies, i.e., plastic deformation was not taken into account in the analysis. The material constants used for the analysis are given in Table 1. In order to clarify simply the distribution characteristics of the residual stress generated near the Y₂O₃ particle, analysis models of the Y₂O₃ particle shape that were investigated in this analysis were circular or square. The interaction between particles is not taken account in this analysis, and so the plate with a central particle is ten times as wide as the Y₂O₃ particle.

The analysis results of residual stress distribution (\( \Delta T = 1000 \) K) generated in the circular and square vicinity of Y₂O₃ particle are shown in Figs. 7(a) and (b), respectively. The residual stress of the required sintering temperature can be calculated easily using the elastic analogy. It is clear that high tensile stress can be observed inside the Y₂O₃ particle. when the sintering temperature is above 2000 K, the very high residual stress is generated inside the Y₂O₃ particles. In the case of the circular Y₂O₃ particle, the residual stress is uniform inside the Y₂O₃ particle and high compressive stress can be observed in the W matrix around the Y₂O₃ particle. In the case of the square Y₂O₃ particle, high stress concentration can be observed at the corners of the Y₂O₃ particle in comparison with the circular Y₂O₃ particle. The authors have previously calculated that the four point bending strength at room temperature for the pure sintered Y₂O₃ is 62.3 MPa. It is clear that the residual stress generated inside the Y₂O₃ particle is higher than the
strength of sintered Y₂O₃. It seems that the cracking of Y₂O₃ particles arises easily during the fabrication process or in operation. Figure 8 shows a typical example of an SEM observation of the Y₂O₃ particle in the Y₂O₃-dispersed W alloy. Some cracking can be seen inside the Y₂O₃ particle. However, the cracking cannot be observed anywhere in the W matrix.

From the above results concerning residual stress characteristics, we consider that the strength of the Y₂O₃-dispersed W alloy can be determined by acting the Y₂O₃ particle as cracks in the W matrix. From this assumption, it is possible to evaluate the strength of the Y₂O₃-dispersed W alloy by use of the fracture mechanics approach\(^\text{(7-10)}\). The relationship of the strength of the Y₂O₃-dispersed W alloy and the average size of Y₂O₃ particles is shown in Fig. 9, as well as the relationship of the strength of the pure sintered W and the average size of pores. The results for both the Y₂O₃-dispersed W alloy and the pure sintered W can be represented by a single line drawing described by Eq. (1), regardless of the sintering temperature and the additive volume of Y₂O₃ particles.
\( \sigma_b = \text{material constant} / \sqrt{a_f} \)  

(1)

where \( \sigma_b \) is the average bending strength and \( a_f \) is the average size of the Y_2O_3 particles. The \( K_{IC} \) value shown in this figure is calculated from the equation \( (\sigma_b = K_{IC} / \sqrt{a_f}) \). This assumes an infinite plate with a crack under uniform tension.

It is found that the Y_2O_3 particle size in the Y_2O_3 dispersed W alloy and the pore size in the pure sintered W produce the similar effect on the strength. It is confirmed that the strength of the Y_2O_3 dispersed W alloy can be easily evaluated by use of the fracture mechanics approach. And in the range for small Y_2O_3 particles, the bending strength is lower than that predicted by Eq. (1), as shown in Fig. 9. This is due to nonlinear fracturing.

5. Conclusions

Strength properties of Y_2O_3 dispersed W alloy were investigated by focusing on the effect of flaw introduction effect due to the dispersed Y_2O_3.

(1) The size of W crystals and Y_2O_3 particles become increases larger with increasing sintering temperature. The growth of W crystals is suppressed with increasing additive volume of the Y_2O_3 particles and the size of the Y_2O_3 particles become increases larger.

(2) The relative density increases with increasing sintering temperature. This tendency becomes more marked with the addition of the Y_2O_3 particles. However, the four point bending strength decreases with increasing the sintering temperature. This tendency also becomes more marked with increasing addition of the Y_2O_3 particles.

(3) Finite element analysis confirms that the residual stress inside the Y_2O_3 particles during the fabrication process leads to the generation of high tensile stress. In addition, the fracture model of the Y_2O_3 dispersed W alloy suggested that the cracking of the Y_2O_3 particles acted as defects in the W matrix.

(4) It is confirmed that the strength of the Y_2O_3 dispersed W alloy and the pure sintered W can be evaluated by use of the fracture mechanics approach. In other words, it can be assumed that both the Y_2O_3 particles in the W alloy and the pores in the sintered W act as cracks.

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References


