OS04F072

Thermal Stress Measurement of Tungsten fiber Reinforced Titanium by X-ray Diffraction

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Abstract

Titanium is one of the useful materials in industrial fields because of the good properties of light weight, high temperature resistance and so on. In this study, the tungsten fiber reinforced titanium composite (W/Ti) was produced by the spot welding method. This manufacturing method used only a simple spot welding system, and it did not need a vacuum chamber and a high temperature furnace such as existing common methods. The arranged tungsten fibers were held between titanium plates (thickness 0.5mm) and fixed by the spot welding. Therefore, this W/Ti composite produced by the spot welding did not joint in the whole position between the tungsten fiber and the titanium matrix because of the partial welding in spot welding points. The coverage, a rate of welding area to the whole plate area, became in 100% for the sample in this study, because it should make up for the partial welding by this method. X-ray stress measurement is the most useful non-distractive method for estimating residual stresses on material surface. It is inferred the W/Ti sample included thermal stresses generated from the thermal expansion coefficient between the titanium matrix and the tungsten fiber. These thermal stresses of the W/Ti composite were measured by x-ray stress measurement. Furthermore, the alteration of thermal residual stress under the thermal cycling was measured by the in-situ x-ray stress measurement technique. These results were discussed from the viewpoint of the thermal expansion coefficient between fiber and matrix.

Key words: W/Ti composite, Spot welding, X-ray stress measurement, Thermal stresses, In-situ stress measurement

1. Introduction

Fiber-reinforced materials have found considerable application in the improvement of the performance characteristics of engineering materials. Commonly, there is a large difference in thermal expansion coefficients between the matrix and the fiber, which causes thermal stress development in a composite system ^(1,2). The effective method has been made on analyzing the thermal stress on the surface of composite materials by the x-ray stress measurement technique ^(2,3). This method is essentially based on the $\sin^2\psi$ method ⁽⁴⁾.

In this study, thermal residual stresses in the continuous tungsten fiber reinforced

titanium matrix composite (W/Ti composite) were examined by the x-ray stress measurement method. This W/Ti composite was produced by the simply spot welding method. Furthermore, stress alterations during high temperature cycling were estimated in the W/Ti composite by the combination of x-ray diffraction and a high temperature vacuum furnace system ⁽³⁾. Temperature is changed from 15°C to 700°C periodically, and thermal stresses in the tungsten fiber and the titanium matrix were measured at each temperature.

2. Preparation of Fiber Reinforced Material

A fiber reinforced metal matrix composite was produced for the present investigation. In this composite, 99.9 % purity tungsten fiber with 100 μm diameter and 99.9 % purity titanium were used for the fiber phase and the matrix phase, respectively.

In this study, the tungsten fiber reinforced titanium composite (W/Ti) was produced by the spot welding method. This manufacturing method used only a simple spot welding system, and it did not need a vacuum chamber and a high temperature furnace such as existing common methods. The arranged tungsten fibers were held between titanium plates (thickness 0.5mm) and fixed by the spot welding. Therefore, this W/Ti composite produced by the spot welding did not joint in the whole position between the tungsten fiber and the titanium matrix because of the partial welding in spot welding points. The coverage, a rate of welding area to the whole plate area, became in 100% for the sample in this study, because it should make up for the partial welding by this method.

Fig. 1(a) shows the schematic diagram of the manufacturing method for the W/Ti composite by the spot welding. The tungsten fiber wound on the titanium plate (thickness 0.5mm) was slipped in between the other titanium plates. These three plates whose the center plate was winded the continuous tungsten fiber were welded by the spot welding system. This W/Ti composite produced by the spot welding did not joint in the whole position between the tungsten fiber and the titanium matrix because of the partial welding in spot welding points. Therefore, the spot welding must be overlapped with the next welding point. Fig. 1(b) shows the spot welding positions which define the coverage for W/Ti composite. The coverage, a rate of welding area to the whole plate area, became in 100% for this sample in Fig. 1(b).

Fig.1 Schematic diagram of: (a) the manufacturing procedure by the spot welding method, (b) the spot welding positions which define the coverage for W/Ti composite.

Fig.2 shows photographs of the W/Ti composite. Fig.2 (a) shows a lot of indentations by the spot welding were observed on the sample surface. Fig.2 (b) is the microphotograph of the cross section of W/Ti composite. The distance between the tungsten fibers is about 0.5mm. Fig.2 (c) is the specimen surface. This surface was polished by #1500 emery paper. The conditions of the spot welding were shown in Table 1.

Fig.2 Photographs of the W/Ti composite; (a) a lot of indentations by the spot welding were observed on the sample surface, (b) a microphotograph of the cross section of W/Ti composite, (c) the specimen surface polished by #1500 emery paper.

Table 1 Conditions of spot welding.

Welding voltage & current	200V, 7.8A
Welding pressure	1.9kN
Holding time	200msec.
Diameter of electrode	11mm

After the spot welding, the surface of the specimen was polished with #1500 emery paper in order to strip off the indentations of the spot welding. Therefore, the surface of W/Ti sample has residual stresses caused by emery polishing. These residual stresses were estimated by the x-ray stress measurement. The final dimension of the sample for the in-situ thermal residual stress measurement was 18mm × 15mm, thickness 1.5mm. The volume fraction of the tungsten fiber is about 5 % in this sample.

3. Theory of X-ray Stress Measurement

The long-fiber reinforced composite considered in this investigation is schematically

shown in Fig. 3. The x_1 and x_2 axes are parallel to and the x_3 axis is normal to the specimen surface, with the x_1 axis being defined as parallel to the fiber direction. The normal lattice strain $\varepsilon_{\phi\psi}$ of a particular hkl plane defined by the angles ϕ and ψ is also shown in this figure.

Fig.3 Coordinate system and definition of the ψ and ϕ angles.

According to the theory of isotropic elasticity, the strain $\varepsilon_{\phi\psi}$ is given by the following equation:

$$\begin{aligned} \varepsilon_{\phi\psi} = & \frac{1+\nu}{E} (\sigma_{11} \cos^2 \phi + \sigma_{12} \sin 2\phi + \sigma_{22} \sin^2 \phi - \sigma_{33}) \sin^2 \psi + \frac{1+\nu}{E} \sigma_{33} \\ & - \frac{\nu}{E} (\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1+\nu}{E} (\sigma_{23} \sin \phi + \sigma_{31} \cos \phi) \sin 2\psi, \end{aligned} \quad (1)$$

where E is Young's modulus, ν is Poisson's ratio. To consider the longitudinal stress σ_{11} parallel to the fibers, we define the angle ϕ equals zero. It was previously reported that both the stresses parallel normal to the fiber are principal stresses and no shearing stress exists⁽²⁾. Therefore, the Eq. (1) is transformed into Eq. (2) for $\phi=0^\circ$.

$$\varepsilon_{\phi=0,\psi} = \frac{1+\nu}{E} (\sigma_1 - \sigma_3) \sin^2 \psi + \frac{1+\nu}{E} \sigma_{33} - \frac{\nu}{E} (\sigma_1 + \sigma_2 + \sigma_{33}). \quad (2)$$

If the plane stress condition is maintained in the surface layer, we can assume the stress $\sigma_3=0$ and Eq.(2) change to following equation.

$$\varepsilon_{\psi} = \frac{1+\nu}{E} \sigma_1 \sin^2 \psi - \frac{\nu}{E} (\sigma_1 + \sigma_2). \quad (3)$$

From this equation, it means that the relationship of ε_{ψ} vs. $\sin^2 \psi$ is linear, and the longitudinal stress σ_1 is determined from the slope of the ε_{ψ} - $\sin^2 \psi$ diagram. Therefore, the $\sin^2 \psi$ method can be used for the estimation of the stress condition in surface region.

Furthermore, when ε_{ψ} is calculated from the diffraction angle $2\theta_{\psi}$,

$$\varepsilon_{\psi} = \frac{d_{\psi} - d_0}{d_0} = -(\theta_{\psi} - \theta_0) \cot \theta_0 \cdot \frac{\pi}{180}, \quad (4)$$

where θ_0 (degrees) is the diffraction angle of the stress free state. The 2θ - $\sin^2 \psi$ diagram can be obtained by measuring 2θ at several values of ψ angles. By substituting Eq.(4) into Eq.(3), we obtain the following.

$$\sigma_1 = -\frac{E}{2(1+\nu)} \cot \theta_0 \frac{\partial(2\theta_{\psi})}{\partial(\sin^2 \psi)} \cdot \frac{\pi}{180} \quad (5)$$

The gradient of the regression line in this 2θ - $\sin^2 \psi$ diagram is equal to the term $\partial(2\theta)/\partial(\sin^2 \psi)$ in Eq. (5).

4. In-situ Thermal Stress Measurement

In this study, stress alterations caused from high temperature cycling was measured by the in-situ x-ray stress measurement method. The high temperature vacuum furnace was mounted on the diffractometer system. The sample of the W/Ti composite was installed on the sample holder in the vacuum furnace. In this measurement, the thermal residual stresses σ_1 parallel to the longitudinal direction, see in Fig.1, of the tungsten fiber were measured in the tungsten fiber and titanium matrix, respectively.

Fig. 4 shows the schematic diagram of temperature vs. time program for in-situ stress measurement of W/Ti composite. The temperature program was controlled by PID temperature controller automatically. Temperature was repeated three times from 15°C to 700°C in case of the tungsten fiber. On the other hand, the temperature change was from 15°C to 550°C in case of the titanium matrix, because the titanium oxide was generated on the sample surface over 600°C.

Fig.4 Temperature programs of in-situ stress measurement for; (a) tungsten fiber, (b) titanium matrix in W/Ti composite.

The rate of temperature change is about 5°C /min in heat up stage and 2°C /min in cool down stage. When the temperature came to a target position, it was holding for about 10 minutes in order to stabilizing, and after that the stress measurement started in every case. The measuring time for x-ray stress measurement in each temperature is about 40 minutes for the tungsten fiber and 110 minutes for the titanium matrix.

Table 2 conditions of stress measurement.

Characteristic x-rays	CuK α
X-ray optics	Parallel beam
Tube voltage	40 kV
Tube current	25 mA
$\sin^2 \psi$	1.0, 0.5
hkl plane & Diffraction angle	W 321, $2\theta=131.1^\circ$ Ti 213, $2\theta=139.3^\circ$
2θ step angle & fixed time	W: 0.2° , 40sec. Ti: 0.1° , 90sec.
Filter	Nikkel
Irradiated area	10×5 mm
Peak deciding method	HWHM method

Table 2 shows the conditions of the in-situ x-ray stress measurement. In this measure, only two points of the ψ angle (0° and 45°) were employed, because the sash bar of the furnace window was cut the x-ray beam.

The intensity of diffraction beam was very weak because the diffraction beam passed through the window of the vacuum furnace. Especially, the diffraction profile from the titanium matrix included the high level back ground because the fluorescence x-ray was generated in the titanium sample. Therefore, we had to keep long fixed times in each 2θ step in order to the accurate and clear measurement. The fixed time is 40 seconds for the diffraction from the tungsten fiber and 90 seconds for the titanium matrix.

5. Results and Discussions

Fig.5 shows the x-ray diffraction profile of W/Ti composite. The diffraction peaks of tungsten fiber and titanium matrix were appeared clearly. The marks $\circ$ and $\bullet$ in Fig.5 are diffraction peaks from the tungsten fiber and the titanium matrix, respectively. The diffraction peaks of W 321, $2\theta=131.1^\circ$ and Ti 213, $2\theta=139.3^\circ$ were employed in this x-ray stress measurement.

Fig.5 Diffraction profile of W/Ti composite. The tungsten fiber exposed on the surface of W/Ti composite by #1500 emery polished.

From Fig.5, the diffraction profile from W 321 overlapped a small titanium peak. However, this peak was ignored for the measurement because of the smallness intensity comparing the tungsten peak. On the other hand, the textile structure was not observed in both of the tungsten fiber and the titanium matrix. Therefore, the peak intensity from the both materials was enough to measure the thermal residual stresses in the every ψ tilt angles.

Fig.6 and Fig.7 show results of the in-situ thermal stress measurement against the longitudinal direction stress σ_1 in the tungsten fiber and the titanium matrix, respectively. The result of the first heat up stage is plotted by the dotted line in red. The blue lines are the result of the thermal cycle stage after the first heat up stage. From these results, the initial residual stress in tungsten fiber is compressive state about -820MPa and titanium matrix is

also compressive about -290MPa. It is supposed that the both of compressive residual stress states in tungsten fiber and titanium matrix were generated by the emery polishing in the initial treatment for the W/Ti composite.

From the result of tungsten fiber in Fig.6, the initial residual stress decreased gradually with the temperature raising under the first heat up stage, and it changed to about -10MPa in 550°C. These thermal stresses are generated from a difference of the thermal expansion between the tungsten fiber and the titanium matrix.

Fig.6 Measurement results of the thermal stress alteration in tungsten fiber under the thermal cycling from 15°C to 700°C. The result of the first heat up stage is plotted the dotted line in red. The blue lines are the result of thermal cycling after the first heat up stage.

Fig.7 Measurement results of the thermal stress alteration in titanium matrix under the thermal cycling from 15°C to 500°C. The first heat up stage and the other cycling are as well as Fig.6.

In second cool down stage, thermal stresses increasing from -10MPa to -1200MPa with the temperature dropping. Furthermore, in the next heat up and down stage, thermal residual stresses changed through the almost same route of the second cool down stage.

Fig.7 shows the thermal/residual stress alteration of the titanium matrix. The initial compressive residual stress -290MPa was changed to the tensile state 30MPa at 500°C with the first temperature rising. It is assumed that the initial residual stress in compressive state was caused by the emery polish, and this compressive residual stresses were cancelled in the high temperature. On the other hand, the tensile thermal stresses were generated from the thermal expansion mismatch between the tungsten fiber and the titanium matrix in 500°C. In second cool down stage, thermal stresses gradually increase from 30MPa to 45MPa with the temperature dropping. In the next heat cycling stage, thermal residual stresses changed through the almost same route of the second cool down stage.

From Fig.6 and Fig.7, if it is considered the balance of thermal stresses between the tungsten fiber and the titanium matrix, these results may not contradict the stress balance qualitatively, because the thermal stresses of the tungsten fiber and the titanium matrix existed in compressive state and tensile state respectively, except the first heat up stage.

Regarding to these stress alterations in thermal cycling, it is assumed that these phenomena are brought about a difference of thermal expansion between tungsten fiber and titanium matrix.

Fig.8 shows the comparison of the elastic calculation and experimental results in the tungsten fiber and the titanium matrix. In this figure, the continuous lines means the average of experimental results of the thermal cycling after first heat up stage, and the dotted line means the results of simple elastic calculation by the following theoretical equation:

$$\begin{aligned}\sigma_w &= \frac{E_w E_{Ti} V_{Ti}}{E_w V_w + E_{Ti} V_{Ti}} (\alpha_w - \alpha_{Ti}) \Delta T \\ \sigma_{Ti} &= \frac{E_w E_{Ti} V_w}{E_w V_w + E_{Ti} V_{Ti}} (\alpha_w - \alpha_{Ti}) \Delta T\end{aligned}\quad (6)$$

where suffixes mean materials, E is young modulus, α is coefficient thermal expansion, ΔT is temperature difference and V is volume fraction. Table 3 shows parameter of this elastic calculation. The stress free position was fixed on the maximum temperature position at 700°C in the tungsten fiber and at 500°C in the titanium matrix for the sake of convenience.

Table 3. Parameters of elastic calculation.

	Young modulus: E , GPa	T. exp. Coefficient: α , $\times 10^{-6}$ 1/°C	Volume fraction: V , %
Titanium	106.0	8.4.0	95
Tungsten	417.7	4.5×10^{-6}	5

From these results, the calculated thermal stresses of tungsten fiber and titanium matrix exist in the compressive state and the tensile state, respectively. Furthermore, these stresses increase with the temperature change from the high temperature to the room temperature. Therefore, the experimental results and the elastic calculations coincide with qualitatively. However, the experimental stress values were larger than the calculated results in every temperature. Especially, the stress value of tungsten fiber at 15°C is -1200MP in experimental result. This value is too large for the case of the volume fraction 5% in the calculated value. If the volume fraction of tungsten fiber is assumed in 0.5%, the calculated stress value becomes -1150MPa which is near the experimental stress value -1200MPa.

Fig.8 Comparison of results of the elastic calculation and the experimental for the tungsten fiber and the titanium matrix.

The cause of these inconsistencies between both stresses is not found as yet, and this is one of the important problems in future investigation.

5. Conclusions

1. The tungsten fiber reinforced titanium composite was manufactured by the spot welding method.
2. Regarding to the initial residual stress σ_1 in fiber longitudinal direction, the compressive stress existed in the both of the tungsten fiber and titanium matrix.
3. Thermal residual stresses σ_1 in tungsten fiber and titanium matrix were changed to other states depending on temperature changes.
4. The main factor of thermal stress alteration is difference of thermal expansion between tungsten fiber and titanium matrix.

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