Applicability of Potassium Titanate Fiber to Metal Matrix Composites*

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The present paper deals with the reaction of the potassium titanate fiber on four kinds of pure metals (aluminum, nickel, titanium and copper) in air and vacuum at elevated temperatures, in order to find the applicability of the fiber to MMC. Results obtained are summarized as follows: (1) The fiber does not react on the aluminum matrix up to the melting point of the matrix. Beyond the point, however, it is found by EPMA line analysis that potassium in the fiber disappears. (2) Up to 1123 K, there is no detectable reaction between the fiber and the nickel matrix. (3) The fiber does not react in vacuum on both titanium and copper matrices below the melting point of them, while a violent reaction between the fiber and the matrices occurs in the atmosphere at temperatures higher than 1023 K. The above mentioned results suggest that the potassium titanate fiber can be applied as a reinforcement in the aluminum and nickel matrices when the MMC is fabricated in air, as well as in the titanium and copper matrices when fabricated in vacuum.

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I. Introduction

Metal Matrix Composites have been considerably attracting in the materials field because of their potential application to mechanical parts. Various inorganic fibers such as glass fiber(1)(2), borsic oxide glass fiber(3)(4), copper-precipitated devitroceramic fiber(5)(6), boron or borsic fiber(7)(9), carbon fiber(10)(13), silicon carbide fiber(14)(16) and aluminum oxide fiber(17)(19) have been developed as reinforcements.

Potassium titanate fiber(20), which has been recently developed, may be expected as a reinforcement to the metal matrices, as it has high tensile strength and is inorganic short fiber. However, either the applicability as a reinforcement of the fiber for metal matrix composite or the suitable fabrication process to metal matrix composite with this fiber is not well established yet. In this paper, the high temperature-stability tests of the fiber with four kinds of pure metals (aluminum, nickel, titanium and copper) in air and vacuum were carried out to find compatibility to the metals and optimum condition to fabricate the metal matrix composites.

II. Materials Used

1. Potassium titanate fiber

Potassium titanate fiber, having a chemical composition of K₂O·6TiO, is a colorless, transparent and fiber-like or needle-like crystal(22). This fiber is excellent in tensile strength, rigidity and thermal resistance. Its characteristics and SEM photograph are shown in Table 1 and Fig. 1. This fiber can be produced in a large scale by the flux method, but it is too fine to be used for the present experiment. Therefore, fibers were dissolved in molten potassium molybdate (K₂MoO₄) and grown to large fibers 3–10 mm in length and 50–150 μm in diameter, which were used in this experiment. The grown fibers have a cavity in the core (Fig. 2) and a lower tensile strength. However, the specific...
Table 1 Characteristics of the fiber processed on large-scale production.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean length (μm)</td>
<td>20</td>
</tr>
<tr>
<td>Mean diameter (μm)</td>
<td>0.3</td>
</tr>
<tr>
<td>Density (Mg/m³)</td>
<td>3.58</td>
</tr>
<tr>
<td>Melting temperature (K)</td>
<td>1643</td>
</tr>
<tr>
<td>Specific heat (kJ/kg·K)</td>
<td>0.92</td>
</tr>
<tr>
<td>Electric resistance (m·Ω)</td>
<td>3.3 × 10¹³</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>6860</td>
</tr>
<tr>
<td>Young's modulus (GPa)</td>
<td>274</td>
</tr>
<tr>
<td>Acid resistivity at room temp.</td>
<td>stable in 10%</td>
</tr>
<tr>
<td>Alkaline resistivity at boiling temperature</td>
<td>stable in 30%</td>
</tr>
<tr>
<td>Affinity to water</td>
<td>good</td>
</tr>
<tr>
<td>Affinity to toluene</td>
<td>normal</td>
</tr>
</tbody>
</table>

* Reference (21)

Fig. 1 SEM photograph of the fiber.

Gravity, specific heat, melting point and electrical resistance are almost the same as those shown in Table 1.

2. Metal matrix

Powders of aluminum, nickel, titanium and copper used as the metal matrix were commercially available, and their purities, particle sizes, densities, and melting points are shown in Table 2.

Table 2 Powder properties.

<table>
<thead>
<tr>
<th>Powder</th>
<th>Purity (%)</th>
<th>Size mesh</th>
<th>Density (Mg/m³)</th>
<th>Melting point (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>99</td>
<td>300</td>
<td>2.7</td>
<td>933</td>
</tr>
<tr>
<td>Ni</td>
<td>99.9</td>
<td>4~7 μm</td>
<td>8.7</td>
<td>1726</td>
</tr>
<tr>
<td>Ti</td>
<td>99</td>
<td>−350</td>
<td>4.54</td>
<td>1943</td>
</tr>
<tr>
<td>Cu</td>
<td>99.9</td>
<td>300</td>
<td>8.96</td>
<td>1356</td>
</tr>
</tbody>
</table>

Fig. 2 A fiber for the use of reaction test, grown to a large size.

Fig. 3 A fiber for the use of reaction test, grown to a large size.

III. Experimental Method

Fibers were arranged upright in a metal die, into which metal powder was packed. The packing was compressed under a pressure of 780 MPa, and the compact had a diameter of 6 mm and a length of 5 mm. Then, in order to study the reactivity between the fiber and the matrix, the compact was heated in air. The heating temperature and the holding time were set in accordance with the kind of the metal matrix used. In addition, a compact with copper or titanium matrix was heated in vacuum (2.7 mPa) for comparison with results of heating in the atmosphere. A section surface of the sintered specimens, perpendicular to the axis of fibers, was polished and then subjected to microscopic observation and line analysis by an electron probe micro-analyser (EPMA). The copper-matrix specimens were subjected to X-ray powder diffraction analysis for analysis of reaction products.
IV. Results and Discussion

1. Aluminum matrix

(1) Reaction with aluminum

Figure 3 shows the results of the line analysis on a packed specimen with aluminum matrix before heating. This indicates levels of titanium and potassium contained in the original fiber.

Figure 4 shows microphotographs on specimens with aluminum matrix after sintering. The central gray zone indicates the fiber; its surroundings are the aluminum matrix. Black areas in the fiber are cavities; Black parts in the matrix are pores and traces of aluminum particles removed during polishing. A black area at the fiber-matrix interface in Fig. 4(c) indicates a part of the fiber chipped off during polishing. As is found in Fig. 4, the interface between the fiber and the matrix is clearly recognized, and no reaction occurred between them up to a temperature several tens of degree (K) below the melting point of aluminum.

It is important to find a change in the transfer state of component element on samples before and after sintering. A sample with aluminum matrix was held for 3.6 ks at various temperatures and then subjected to line analysis by EPMA. From this result it was found that no change had been recognized in the content and distribution of potassium and titanium in the fibers and the fiber-matrix interface as shown in Fig. 3. Figure 5 shows microphotographs of the fibers which were held in molten aluminum for 3.6 ks at 973 or 1073 K and then cooled. In the sample heat treated at 1073 K, a change can be distinctly seen at the fiber-matrix interface. Figure 6 shows the line analysis of the sample of Fig. 5(b). From comparison of Fig. 6 with Fig. 3, it is clearly found that potassium in the fibers has almost disappeared by holding them at 1073 K.

(2) Compatibility between fiber and metals

The reaction layer formed by reaction between a fiber and the matrix will usually decrease the fiber strength(23)-(25) and reduce the strength of composites(26)-(29). As the reaction layer is more brittle and thicker, the strength reduction will be more significant(23)(24)(26). However, it has been reported that the wettability and adhesion between a fiber and the matrix will be low(25)(30) when
no reaction occurred at the interface. As the reactivity to improve the wettability is so slight, the reaction layer cannot be detected by optical microscope or an EPMA. Therefore, in this experiment, the necessary condition to apply the fiber as the reinforcement was judged to be met, when no change was observed in the composition of the interface and the fiber. Further, it may be said that the sufficient condition can be also met when a reinforcement provides a good wettability and yet little reduction in its strength. However, the study on this problem should be carried out further on a composite with potassium titanate fiber.

From the results and discussion described above, the necessary condition to apply potassium titanate fiber in aluminum matrix can be obtained, when the composite is sintered at a temperature below the melting point of aluminum, because no compositional change was observed in the interface and the fiber. However, care should be taken, when such a composite is to be made by the liquid method at a temperature above the melting point of aluminum, since the composition of component element will be changed in the interface and the fiber.

2. Reaction with nickel

Figure 7 shows microphotographs of potassium titanate fibers in nickel matrix sintered at high temperature in air. The compositional variation at the interface was not observed (the black areas in the fibers show partial removal of fibers during polishing). In addition, this fact is found also in either the interface or the fiber on all samples sintered at any temperatures tested.

From the above results, potassium titanate fiber can be considered to be well applicable as a reinforcement in nickel matrix.

3. Reaction with titanium

Figure 8 shows microphotographs of potassium titanate fibers in titanium matrix heated at various temperatures. As indicated in the figure, a significant change is observed at the fiber-matrix interface in the samples heated above 1073 K. No potassium is left in the fiber in the composite heated at 1073 K for 3.6 ks, as shown in Fig. 9(a). On the contrary, the titanium contents in the fiber increased by 16 and 25% than that in the original fiber in the
composites heated for 3.6 ks at 1073 and 1123 K, respectively. These change are shown by black circles in Fig. 10. The composite, also, was sintered in vacuum, because titanium might react vigorously with oxygen at high temperature. As shown in Fig. 9(b), the fiber in the vacuum sintered composite had the same composition as the unheated fiber. Also, as shown by the open circles in Fig. 10, no change was observed in the composition of fibers, although they were heated in vacuum at various temperatures. Thus, in the vacuum heated samples, both the fiber and the fiber-matrix interface were not changed at all.

As for the potassium titanate fiber-titanium composite, it is not found that the potassium titanate fiber is effective as a reinforcement when the composite is heated at 1073 K in air. On the contrary, it is seen that the fiber has an effective action when the composite is heated in vacuum, because no change in the fiber composition was observed at the temperature tested. Therefore, to fabricate the composites with potassium titanate fiber, the mixture must be heated at a temperature below 1023 K in air, but it must be heated in vacuum at temperatures above 1023 K. In practice, however, the process will not be an easily employed technique to prepare the composite.

4. Reaction with copper

Figure 11 shows microphotographs of potassium titanate fiber in copper matrix, heated at 1023 K for various hours. As shown in the figure, reaction products are formed at the interface. However, the line analysis indicated that the fiber itself had not been changed. CuO and Cu$_2$O were detected in the composites which had been heat treated in air.
Fig. 9 EPMA line analysis of the fiber heated for 3.6 ks at 1073 K in titanium matrix.

and in vacuum with a purpose to identify the reaction products. The line analysis of the heat treated samples in vacuum indicates that the composition of fiber was little changed and no reaction products were also formed at the interface.

From these results, it can be considered that the preparation of the composites in air is not effective to reinforce the copper matrix since copper oxides are formed at the interface and cavities in the matrix.

Table 3 summarizes the results of series of experiments so far described.

V. Conclusions

Reactions between potassium titanate fiber and pure metals (Al, Ni, Ti and Cu) at elevated temperatures in a vacuum or in air were examined, in order to find applicability of the fiber as a reinforcement to metal matrix composites. The results are summarized as follows:

1. The fiber did not react with aluminum in air up to the melting point of the matrix. However, the potassium component in the fiber dissolved into the matrix metal at a high temperature corresponding to the liquid state of aluminum.

2. No detectable reaction of the fiber with nickel was found up to 1123 K in air.

3. The fiber reacted slightly with titanium
or copper in vacuum at a temperature above 1073 K, while remarkable reactions between the fiber and both matrices occurred in air at the temperature.

(4) These results suggest that the potassium titanate fiber is applicable as a reinforcement in aluminium or nickel matrix, when the composites are fabricated in air. In the case of titanium or copper, the composites should be fabricated in a vacuum.

**Acknowledgement**

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**REFERENCES**

(3) R. Thompson, S. Badzioch, R. H. Biddulph, G. K.
Applicability of Potassium Titanate Fiber to Metal Matrix Composites


(15) J. Tanaka: Kogyo Zairyo, 27, 1 (1979), 53.


