Fabrication of a carbon fibre/aluminium alloy composite under microgravity

YOSHINAO MISHIMA, MASASHI HORI*[‡], TOMOO SUZUKI, SOKICHI UMEKAWA[§] Besearch Laboratory of Precision Machinery and Electronics, and *Gradua

Research Laboratory of Precision Machinery and Electronics, and *Graduate School, Department of Materials Science and Engineering, Tokyo Institute of Technology, Nagatsuta, Midori-ku, Yokohama 227, Japan

Fabrication of a composite material with ultra low density and high stiffness under microgravity is the objective of the present investigation. The composite structure to be obtained is a random three-dimensional array of high modulus, short carbon fibres bounded at contact points by an aluminium alloy coated on the fibres. The material is highly porous and thus has a very low density. The motivation toward the investigation, simulation experiments, choice of the component materials and in-flight experiment during ballistic trajectory of a National Space Development Agency rocket are described herein. Supporting experimental evidence shows that the cohesion between the carbon fibre and the aluminium alloy is excellent, by which the achievement of desired properties of such composites seems probable.

1. Introduction

Structural materials in space are desired to possess high specific strength, stiffness and resistance to buckling. Also it is desirable that the materials are capable of in-orbit fabrication. Such requirements could be met, in principle, by a material such as a finely foamed metal-ceramic composite where the macroscopic buckling load can be raised by an increase in the second moment of area of the crosssection at a constant mass, whilst microscopic buckling at the metal walls between small cavities is prevented by fine ceramic particles or fibres dispersed in the metal matrix. Fabrication of such materials was our original proposal for the present the First Materials Processing Test (FMPT) project of NASDA, being scheduled in early 1988 on Spacelab-J. However, preliminary investigations revealed two difficulties to overcome, which are: (a) to confine cavities to an isolated fine state in a molten metal even under a microgravity since metals have generally very low viscosity and very high surface tension in the molten state; and (b) to find a suitable constituent which will act as a foaming agent in a molten metal and is yet safe in carrying out the fabrication.

An alternative method to obtain a similar type of structure which would have the properties stated above was then proposed. The structure consists of short high modulus ceramic fibres and a small amount of filler metal of relatively low density. The fibres are aligned as random three-dimensional arrays and are bonded at contact points by the metal that is coated on the surface of the fibres. Such a structure is highly porous and cavities are surrounded by the metal walls to resemble the structure of the foamy hybrid composite in the original proposal. The fabrication procedure in this case involves fibre coatings with a metal or an alloy, encapsulation of chopped fibres and heating the material to a temperature above the melting point of the coated metal followed by subsequent cooling. It is simple enough and thus has an advantage in considering the possibility of in-orbit fabrication. Microgravity during fabrication is essential for two reasons: (i) keeping short fibres in random three-dimensional configuration under the effect of gravity; and (ii) keeping the molten metal from slipping and separating down along the direction of gravity due to a difference in densities of the fibre and metal.

2. Simulation experiment

In order to provide a better picture of the structure under consideration, results of a simulation experiment are briefly shown. In the experiment, Nylon threads of 0.8 mm in diameter were used as fibres and they are coated with wax to a thickness of some 0.2 mm. The coated threads are chopped into pieces of 5 mm in length and they are heated to temperature for some time during which the wax coatings melt in a Pyrex glass container. To simulate a microgravity environment, coated threads are placed in an aqueous solution of alcohol of which the density was adjusted to be that of the wax coatings. Upon heating and subsequent cooling, the threads are bonded at contact points. Fig. 1a is of the composite obtained under the microgravity simulation and Fig. 1b is that prepared under the effect of gravity. In Fig. 1a, a random threedimensional array of the short threads bonded at

[‡]Present address: Nippon Kokan KK, Minami-Watarida, Kawasaki-ku, Kawasaki 210, Japan. [§]Present address: Science University of Tokyo, Noda-shi, Chiba 278, Japan.



Figure 1 Structure of Nylon threads-wax composites fabricated under (a) microgravity simulation and (b) the effect of gravity.

contact points is achieved, whereas in Fig. 1b the wax was separated from the Nylon threads and was. deposited at the bottom. The difference is clearest when comparing the height of the specimen in three figures, because for each fabrication the same amount of material was used.

3. Choice of materials

A high modulus carbon fibre was chosen for this experiment and an aluminium alloy was developed as a coating substance for the fibres. An aluminiumbased alloy was selected because of its low density and its moderate melting temperature at which fabrication is practically feasible with non-expensive, simple electric furnaces. It is recognized that the wettability between carbon fibre and pure aluminium is well known to be extremely poor. The wettability between them is known to be improved at temperatures above 1273 K due to the formation of aluminium carbide (Al_4C_3) [1, 2]. The carbide formation is known to degrade the fibre and this chemical reaction has been found to occur even at 773 K or lower upon prolonged exposure [1-6]. In the present experiment, therefore, a systematic investigations was undertaken on improvement in compatibility between carbon fibre and aluminium by alloving the aluminium. Aluminium alloys containing a small amount of various alloying elements (up to 5 at %) prepared by arc-melting were deposited on to carbon fibres and the coated fibres were heated to 1073 K in vacuum to melt the alloys. Then after cooling down, the surface of the fibres was examined by scanning electron microscopy (SEM) to judge the wettability.

As is shown in Fig. 2a, the surface of the fibres coated with pure aluminium have a number of metal droplets indicating the poor wettability. It was found that when additions of 1 at % of such elements as lead, thallium and indium were made, the wettability is significantly improved as shown in Fig. 2b for the case of an Al-1 at % Pb alloy. Moreover, tensile tests of the coated fibres after the heating showed that the room temperature strength of the carbon fibres was not deteriorated by the coatings and by the heating at 1073 K. The elements to improve the wettability of aluminium against carbon fibre are the heavy metallic elements, however, the improvement is achieved by the addition of only 1 at % and therefore it would not give any difficulty in fabrication of the low density composites. It should be emphasized here that the development of the wetting reagent is widely appreciated for the fabrication of any type of carbon fibre/ aluminium composite in which the infiltration techniques are preferred. The details of the above investigations are described elsewhere [7].

4. In-flight experiment

An in-flight experiment was carried out in August, 1983 using a TT-500A rocket by NASDA during its ballistic trajectory. The fabrication of the carbon fibre/aluminium alloy composite under microgravity was practiced. The details of the materials preparation and the fabrication are as follows.

Carbon fibres of a high modulus type with approximately 7 to 8 μ m in diameter are coated on the surface with an Al-1 at % Tl alloy to a thickness of $1 \mu m$ using an ion-plating technique in an argon atmosphere. The coated fibres are cut into short pieces of 0.5 to 1.0 mm in length and vacuum encapsulated in a silica tube with an inner diameter of 10 mm. Prior to the encapsulation, a piece of lid made of silica pipe was welded onto the inner wall of the tube to slightly compact the materials. The height of the specimen in the silica tube was then ~ 15 mm. The whole specimen capsule, $\sim 45 \,\mathrm{mm}$ in length as shown in Fig. 3, was then packed in a graphite container and was placed in an electric furnace. The TT-500A rocket provided a microgravity environment for 6 min during its ballistic trajectory and the heat treatment of the specimen was scheduled for this time period. The maximum temperature was scheduled to be 1023 K for 2 min while the melting point of the aluminium alloy is about





Figure 2 Scanning electron micrographs of carbon fibres after heating at 1073 K for 30 min coated with (a) pure aluminium and (b) Al-1 at % Pb alloy.



Figure 3 The appearance of the specimen capsule for the in-flight experiment.

943 K. Unfortunately, temperature control of the electric furnace was unsuccessful and the temperature went up to above 1473 K. The comparison between the scheduled time-temperature profile and the actual profile recorded on a telemeter is shown in Fig. 4.

Examination of the retrieved specimen shows obvious damage on the surface of the constituent fibres and the whole specimen was found quite brittle. This is probably due to the chemical reaction between the fibre and the aluminium alloy coating to form Al_4C_3 and due to the partial evaporation of the aluminium alloys both attributed to the extreme overheating. SEM observations, however, revealed the structure of the specimen to be a random threedimensional configuration of the short fibres and the contact-point bonding was mostly successful making the entire specimen rigid, thereby encouraging future experiment. As is shown in Fig. 5 the aluminium alloy coatings are severely deteriorated, from which any mechanical property evaluation was considered meaningless.

5. Cohesion of the carbon fibre/aluminium alloy interface

One of the important factors to determine mechanical properties of such a composite is the cohesion between the carbon fibres and the aluminium alloy coating. As is mentioned earlier, compatibility between them was found to be excellent in terms of the fibre strength after the coating and subsequent heat treatment at 1073 K. The compatibility here to be considered is the one across the interface, by which the composite strength and resistance for buckling are controlled.

A bulk composite consisting of long carbon fibres and an Al-1 at % Pb alloy, one of the alloys which showed good wettability with carbon fibre, was prepared by remelting the layered carbon fibres and the aluminium alloy thin plates. As a reference material a carbon fibre/pure aluminium composite was also prepared by the same method. Then the composite bars of $5 \text{ mm} \times 5 \text{ mm} \times 55 \text{ mm}$ in dimension was notched on all faces to a depth of 1.0 to 1.5 mm and fractured in a direction perpendicular to the fibre length using an impact test machine. Here it is to be noted that the purpose of the test was to obtain a transverse fracture surface by any means and was not to evaluate the impact energy of the composite quantitatively. Fractographic observations revealed that the fibres are well attached to or embedded in the plastically deformed ductile matrix in the composite with the aluminium alloy as is shown in Fig. 6a. At certain locations where the stress state upon fracture would have been in tension along the radial axis of the fibres, they are torn up as shown in Fig. 6b. Fig. 7 shows the fracture surface of the composite with pure aluminium in which absolutely no sign of cohesion between the fibre and the matrix is observed and the fibres are completely pulled out.

These results assure a strong cohesion between the carbon fibre and the aluminium alloy developed in the course of the present investigation. It provides not only a positive aspect in fabricating the composite under consideration here but also a possibility of fabricating bulk carbon fibre/aluminium composites by a simple infiltration technique.

6. Concluding remarks

The present status of our investigations towards fabrication of a new carbon fibre/aluminium alloy composite under a microgravity has been described. It was shown that a three-dimensional configuration of short carbon fibres can be obtained in which fibres are bonded at contact points by the aluminium alloy coated on the surface of the fibres. The cohesion between the fibres and the aluminium alloy was proved to be strong and therefore achievement of



Figure 4 Scheduled and the actual temperature-time profiles during the in-flight experiment. t_1 = power shut off; t_2 = accelerated cooling on; t_3 = accelerated cooling off; t_4 = start of respin.



Figure 5 Scanning electron micrograph of the composite fabricated on the NASDA rocket.





Figure 6 SEM fractography of the bulk composite of carbon fibre and Al-1 at % Pb alloy.



Figure 7 SEM fractography of the bulk composite of carbon fibre and pure aluminium.

desired mechanical properties such as high stiffness and resistance for buckling seems probable in such a composite having an ultra low density. Evaluation of mechanical properties of the composite is the most important current problem. The factors which must be taken into account are the thickness of the aluminium alloy coating, heating temperature and duration, length of the short fibres and optimization of final density for the best mechanical properties.

Acknowledgement

This investigation was supported by the National Space Development Agency and also by Special Coordination Funds for Promoting Science and Technology through the Science and Technology Agency of Japanese Government.

References

- 1. C. MANNING and T. GURGANUS, J. Amer. Ceram. Soc. 52 (1969) 115.
- 2. S. RHEE, ibid. 53 (1970) 386.
- 3. A. A. BAKER and C. SHIPMAN, Fibre Sci. Technol. 5 (1972) 285.
- 4. G. BLANKENBURGS, J. Aust. Inst. Met. 14 (1969) 26.
- 5. P. W. VACKSON, D. M. BRANDDICK and D. J. WALKER, Fibre Sci. Technol. 5 (1972) 219.
- 6. S. J. BAKER and W. BONFIELD, J. Mater. Sci. 13 (1978) 1329.
- 7. Y. KIMURA, Y. MISHIMA, S. UMEKAWA and T. SUZUKI, *ibid.* 19 (1984) 3107.

Received 19 August and accepted 3 October 1985