Fracture and Mechanical Properties of P100 Gr/6061 AI Composite

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The effects of heat treatments on the mechanical and fracture properties of continuous fiber Pitch 100 graphite/6061 aluminum (PI00 Gr/6061 AI) composite were evaluated by varying solution temperature and aging time. All heat treatments resulted in decreased mechanical properties due to the reaction at the fiber/matrix interface during solution treatments. SEM analysis of the fractured samples showed a large degree of fiber pullout, interface failure, and matrix cracking.

1. Introduction

IN recent years, metal-matrix composites have been receiving considerable attention as lightweight materials for use in many advanced technology applications. Metal-matrix composites are produced in various forms, including continuous and discontinuous fiber-reinforced composites. Continuous fiber composites offer exceptional directional specific strength and stiffness. Their dimensional stability and low axial thermal expansion coefficients make them promising materials for missile, aircraft, spacecraft, and automotive components. For example, continuous fiber composites are used in large deployable antennas and booms in spacecrafts. $[1-4]$

Metal-matrix composites are defined as materials containing reinforcements in a continuous metal matrix. These reinforcements may include any combination of fibers, wires, single-crystal whiskers, polycrystalline flakes, and nonmetallic particles. [4-5] The use of a continuous fiber results in a material that is greatly strengthened along the fiber axis.

Fibers in composites are generally unidirectional, but can be patterned in layers with varying orientations. Continuous fiber composites possess very high specific strengths and stiffnesses. In addition, the properties of these composites can be customized. For example, the coefficient of thermal expansion of certain graphite/aluminum composites is zero in the fiber direction. Although continuous fiber composites are generally expensive, they are used in lightweight applications in which directional strength and stiffness are critical. The material examined in this investigation was a unidirectional pitch 100 graphite fiber-reinforced 6061 aluminum composite (P 100 Gr/6061 A1). This material was manufactured by liquid metal infiltration of graphite fiber bundles, followed by hot pressing to form composite sheets.

One of the major disadvantages of continuous fiber composites is poor transverse strength. In various studies, [6-10] heat treating graphite/aluminum has resulted in increased transverse strength. This is attributed to both matrix strengthening and interfacial reaction zone growth. The reaction zone tends to bond the fibers to the matrix. Unfortunately, the reaction zone growth that occurs during heat treatment is detrimental to the mechanical properties in the longitudinal direction.

2. Experiments

The P100 Gr/6061 A1 composite was manufactured by Materials Concepts Inc., Columbus, Ohio. The fiber volume percent was approximately 45%. In Fig. 1 and 2, typical cross sections of the as-received material are presented. Defects such as fiber/fiber contacts and uninfiltrated zones are evident. In previous studies, these defects have been shown to degrade the transverse strength of continuous fiber graphite composites.^[11] The estimated mechanical properties are:

The longitudinal specific stiffness and strength of continuous graphite fiber composites are exceptional. In addition, their coefficients of thermal expansion and many mechanical and physical properties can be customized.

Fig. 1 Microstructure of a P 100 Gr/6061 AI composite sample in the as-received condition showing a tow and an aluminum foil. At the center of the tow, an uninfiltrated zone is present.

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The 2.54-mm (0. l-in.) PI00 Gr/6061 AI sheet was cut into 127-mm by 12.7-mm $(5-$ by 0.5-in.) specimens. Six of the specimens remained in the as-received condition, and the other six specimens were annealed for further heat treatment. The annealing cycle was as follows. The material was heated to 371° C (700 °F), held for 2 hr, cooled to 204 °C (400 °F) at 28 °C/hr (50 ~ and then cooled to room temperature (the cooling rate is not important).

A Model 2223 Clambshell-type furnace from Marshell Furnace Co. Inc. was used for the solution heat treatment. Connected to the furnace was a Cooke Vacuum Products Inc. Model CVE301L Cryogenic Coating Apparatus. Samples were encased in fused quartz tubes before heat treatment. The procedure of the solution heat treatment is as follows. The material was heated to the solution temperature and held at that temperature for a specified time, then water quenched to room temperature. After the solution treatment, the specimens were aged with an Applied Test System, Inc. Series 2911 furnace.

The tension tests were performed using a closed loop servohydraulic Instron Testing Machine, Model 1331, with a 49000- N load cell. A Zenith Data Systems Z-100 with an analog-todigital board was implemented to collect data during the tension test. A computer program modified by Beachtle^[12] was used to record the strain and load output during tension tests. ASME Standard D 3552-77-C was adopted for tension test sample configuration.^[13] Torr Seal, a low-vapor pressure adhesive made by Varied, was used as the sample/end tap adhesive.

Fig, 2 Microstructure of a P100 Gr/6061 AI composite sample in the as-received condition showing fiber/fiber contacts, uninfiltrated zones, and an aluminum foil.

Fig. 3 A strain gage mounted on a P100 Gr/6061 AI sample.

A sensitive strain-measuring method was required because of the small elongation to failure of the continuous fiber composites. Micro-measurements Division Precision Strain Gages, type CEA- 13-125UN-350, were used for strain measurements (Fig. 3). The tension tests were run in the Instron machine with a load range of 100% and a stroke rate of 0.5 mm/min.

The microstructural analysis performed on the samples consisted of optical microscopy, image analysis, scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). To prepare the samples for SEM and EDS, fractured samples were cut approximately 13 mm from the fracture surface using a silicon carbide cut-off saw. These samples were cleaned in an ultrasonic cleaner using acetone, methanol, and trichloroethane, consecutively.

3. Results and Discussion

Figure 4 shows the individual tows used to make this material. The mechanical properties for both the as-received and the heat treated samples are presented in Table 1. For the as-received samples, the tensile strength ranged from 440 to 590 MPa. This large range in values may be due to the large variations in the quality of the as-received material. Because this composite was much thicker than usual (five piles of tows), the samples cut from the edges of the composite sheet displayed very poor bonding between the tows and the aluminum foils. Sample No. 3 was cut from the edge of the composite sheet and had a tensile strength of only 256 MPa. The degree of bonding observed during microstructural analysis varied greatly among the nonedge samples, presumably because of their varied proximity to the edge. These differences in sample quality may have accounted for the tensile strength variation.

According to the rule-of the-mixtures, the tensile strength of P100 Gr/6061 AI is about I000 MPa, assuming that the matrix strength is approximately that of annealed 6061Al. The highest experimental value for the as-received samples was 593 MPa. Theoretical overestimate of the tensile strength of unidirectional composites are common because several factors are not

Fig. 4 Micrograph of an as-received, polished $P100$ Gr/6061 Al cross section.

Table 1 Mechanical Properties

(a) Heat treatment key: solution temperature $({}^{\circ}C)/$ solution time (hr)/age temperature $({}^{\circ}C)/$ age time (hr). (b) End tab adhesive failed during the test.

Fig. 5 SEM photograph of the fracture surface of sample #12 showing fiber bundles.

taken into consideration. First, the mechanical properties of graphite fibers are degraded during fabrication, due to reaction of graphite with aluminum at high temperatures. Second, even with recently developed fiber coatings, poor bonding between graphite and aluminum usually occurs. Third, manufacturing defects such as fiber misalignment, fiber/fiber contacts, and fiber distribution are not taken into consideration.

The experimental elastic moduli of the as-received samples ranged from 300 to 365 GPa. The range of experimental values was probably caused by the same factors described previously for the variation in tensile strength. According to the rule-ofmixtures, the elastic modulus of this material is 352 GPa. Thus, the experimental results were close to the theoretical value, as is common for continuous fiber composites.

All of the heat treatments resulted in lower mechanical properties. After only 1 hr at a solution temperature of 530 \degree C, the tensile strength was reduced to 336 MPa for sample No. 7. Increased time and temperature during solution treatments decreased the mechanical properties further. At solution tempera-

Fig. 6 SEM photograph of the fracture surface of sample #12 showing matrix cracking.

tures of 550 and 570 \degree C, the tensile strength and elastic modulus decreased sharply. These temperatures correspond to the occurrence of carbide formation, as documented in previous TEM studies.^[6,10,11] The reactions that produce carbides result in pits on the fiber surface, which degrade the mechanical properties of the fiber.

SEM analysis of fractured P100 Gr/6061 A1 samples revealed many fracture surface features. In Fig. 5, the fracture of sample No. 12 is shown. This sample was solutionized at 570 ~ for 100 hr. In the bottom left comer, an aluminum foil is evident. The fiber bundles, or tows, fractured individually. In some tows, the fibers pulled out, whereas in other tows the fibers fractured predominantly on the fracture surface. Fiber pullout is characteristic of poor wetting of the graphite fibers by the aluminum matrix during fabrication. Increased fiber pullout may result from reaction of the fiber with the matrix during heat treatment. The reaction between the fiber and the matrix produces carbides at the fiber/matrix interface, as documented in previous studies.^[6,10,11]

In Fig. 6, the surface of a tow in which the fiber fractured predominantly at the fractured surface is shown. Matrix cracking is prevalent in the aluminum-rich zones. Interface failure is also quite common. The fracture path in this sample was along the fiber/matrix interface and through the matrix itself. The fracture path is evident in the top center of the photograph, where the matrix cracks from paths between locations on the fiber/matrix interface. In the upper right comer, a region of touching fibers is pulled out from the matrix. Fiber/fiber contacts were prevalent in optical microscopy photographs of the as-received specimens. In previous studies, touching fibers have been shown to act as fracture initiation sites during transverse loading. [9] During longitudinal loading, touching fibers would also be detrimental, because load transfer to the matrix would be impaired.

Fig. 7 SEM photograph of the fracture surface of sample #12 showing fiber/fiber fracture and interface failure.

In Fig. 7, fracture at the matrix/fiber interface is shown. This figure also provides a closer view of matrix cracking, along with an axially cracked fiber. During optical microscopy, some axially cracked fibers were observed in the as-received material. In Fig. 8, a SEM fractograph of sample No. 11 is presented. This sample was solutionized at 550 $^{\circ}$ C for 72 hr. Individual tows are again evident on the fracture surface. Fiber pullout, however, was less common than in sample No. 12. Because of the lower solution temperature, less degradation of the fiber/matrix interface occurred. According to previous TEM studies, increased carbide formation occurs at temperatures above 550 \degree C. Therefore, the bond between the fiber and the matrix may have been stronger for sample No. 11 than for sample No 12, because less carbides were present at the fiber/matrix interface.

Fig. 8 SEM photograph of the fracture surface of sample #11 showing fiber bundles.

Fig. 9 SEM photograph of the fracture surface of sample #11 at 25 ~ showing fiber pullout.

Fig. 10 SEM photograph of the fracture surface of sample #11 at 25° showing the imprints left by fibers in the aluminum matrix.

Fig. 11 SEM photograph of the fracture surface of sample #11 showing fiber fracture.

In Fig. 9, the fiber pullout is shown from a 25° angle. An enlargement of this photograph is shown in Fig. 10. Ridges of aluminum imprinted by pulled out fibers are evident. No aluminum was observed on the graphite fibers, indicating that the aluminum did not wet the graphite well during fabrication. This phenomenon has been documented in previous stud i es.^[9]

In Fig. 11, a closeup of the fracture surface of sample No. 11 is shown. Interface failure is less evident in sample No. 11 than in sample No. 12. The fracture path traveled through the fibers, rather than through the fiber/matrix interfaces. Again, two touching fibers are pulled out together.

In the two samples examined above, the effect of carbide formation at temperatures above 550 $^{\circ}$ C was evident. For the sample solutionized at 570 $^{\circ}$ C, the fracture path shifted from propagating through the fibers to propagating around the fibers through the fiber/matrix interface. Therefore, the mechanical properties were decreased because the full strengthening effect of the fibers was not utilized.

4. Conclusions

For P100 Gr/6061 AI, heat treatment results in decreased mechanical properties. At solution temperatures in excess of 550 $^{\circ}$ C, severe decreases in the mechanical properties of P100 Gr/6061 AI occur. At these temperatures, the fracture path shifts from through the fibers to through the fiber/matrix interface.

References

- 1. R. DeMeis, *Aerospace America, 26,* Mar (1989).
- 2. D.L. Hunn, *Proc. Symp. Mechanical Behavior of Metal Matrix Composites,* Dallas, Feb 1982, J.E. Hack and M.F. Amateau, Ed., The Metallurgical Society of AIME, 83 (1983).
- 3. B .J. Maclean and M.S. Misra, *Proc. Symp. Mechanical Behavior of Metal Matrix Composites,* Dallas, Feb 1982, J.E. Hack and M.F. Amateau, Ed, The Metallurgical Society of AIME, 301 (1983).
- 4. D. Webster, *Advances in Composite Materials: Proceedings of the Third International Conference on Composite Materials,* Paris, Aug 1982, A.R. Bunsell *et al.,* Ed., Permagon Press, Oxford, 1165 (1980).
- 5. C.G. Krishnadas Nair, M.R. Krishnadev, and D. Dutta, *32nd International SAMPE Symposium and Exhibition,* Anaheim, Apr 1987. R. Carson *et al.,* Ed., Vol. 32, Society for the Advancement of Material and Process Engineering, 889 (1987).
- 6. L.J. Fu, M. Schmer/ing, and H.L. Marcus, *Composite Materials: Fatigue and Fracture, A Symposium on High Modulus Fibers and Their Composites,* Dallas, Oct 1984, H.T. Hahn, Ed., ASTM, 51 (1984).
- 7. J. England and I. W. Hall, *Scripta Metall., 20,* 697 (1986).
- 8. M. Vedula and R.A. Queeney, *Proceedings of a Symposium on Interfaces in Metal-Matrix Composites,* New Orleans, Mar 1986, A.K. Dhingra and S.G. Fishman, Ed., The Metallurgical Society of AIME 227 (1986).
- 9. A. Okura, J. Inagaki, E. Nakata, S. Ikegami, T. Ohsaki, and M. Yoshida, *Advances in Composite Materials: Proceedings of the Third International Conference on Composite Materials,* Paris, Aug 1982, A.R. Bunsell *et al.,* Ed., Permagon Press, Oxford, 1075 (1980).
- 11. J. Lo, D. Finello, M. Schmerling, and H.L. Marcus, *Proceedings of a Symposium on Mechanical Behavior of Metal-Matrix Composites,* Dallas, Feb 1982, J.E. Hack and M.F. Amateau, Ed., The Metallurgical Society of AIME, 77 (1983).
- 10. T. Erturk, J.A. Cornie, and R.G. Dixon, *Proceedings of a Symposium on Interfaces in Metal-Matrix Composites,* Dallas, Feb 1982, J.E. Hack and M.F. Amateau, Ed., The Metallurgical Society of AIME, 89 (1983).
- 12. D. Beachtle, "The Effect of Stress Whitening on Moisture Diffusion in Thermosetting Polymers," M.S. Thesis, Clarkson University, Potsdam, New York (1989).
- 13. ASME Standard D 3552-77, ASME (1988).