Fracture Behavior and AE Signals in Carbon Fiber Reinforced Aluminum

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The correlation between the mechanical property change and the AE signal produced during the 3-point bending test was investigated for the carbon fiber reinforced aluminum. The results of the AE measurement reflect the change of strength by heat-treatment and SEM fractography. The change of the fracture mechanism due to the thermal history of the specimen can be detected by the frequency spectrum analysis of the AE signal. The slight change in the bonding between carbon fiber and aluminum matrix which is not observable with SEM can be detected by the analysis of the AE frequency spectrum diagrams.

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I. Introduction

Recently, the carbon fiber reinforced plastics (CFRP) has been widely utilized for many purposes because the CFRP has the highest specific tensile strength. However, the composites with plastic matrix cannot retain their strength at temperatures higher than 500 K. The development of the high strength composites for elevated temperature use at much higher than 500 K are strongly desired now. The metal matrix composites reinforced by fibers (FRM) such as carbon fiber, alumina fiber and silicon carbide fiber, have been considered to be candidate materials for this requirement. Among them, the carbon fiber reinforced aluminum (CFR-Al)\(^{(1)-(5)}\) is regarded as the one of the most hopeful materials due to the lightness of both components.

However, the combination of these materials is inferior in the compatibility\(^{(6)-(7)}\), and the poor wettability of CF with molten aluminum makes it difficult to obtain stronger bonding during the fabrication of composite. In spite of the poor wettability, the very brittle and deliquescent compound, Al\(_4\)C\(_3\), is easily formed at the interface of these components\(^{(8)-(9)}\) during the fabrication and the application to high temperature use. The most important problem for the production of CF is how to control to minimize the formation of the compound and to detect their formation at earlier stages. The acoustic emission phenomenon has been applied\(^{(10)-(15)}\) to detect the change in the fracture process of the fiber reinforced composites.

In the present work, the correlation between the signal of the acoustic emission (AE) and the change of the mechanical property and fracture mechanism was investigated from the viewpoint of the change of bonding or compatibility.

II. Experimental Procedures

1. Carbon fiber reinforced aluminum composites

The specimens used were the composites fabricated by the hot pressing of the aluminum-preformed carbon fiber (CF). The as-received sample is 50 mm long, 8 mm wide and 2.5 mm in thickness with long CF of about 7 \(\mu\)m diameter aligned parallel to the specimen length and the volume fraction of CF is about

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40 vol%. The specimens for the 3-point bending test were prepared from these blocks by cutting into pieces 22 mm long and 1.7 mm wide by a diamond blade. The bending load was applied to the original top surface of the as-received specimen.

2. Heat treatment for specimens

In order to yield the property change in the specimens such as the degradation of strength expected in high temperature use or in the fabrication processing, the specimens were heated at various temperatures ranging from 670 to 890 K for 3.6 ks in a quartz tube evacuated by a rotary pump. To minimize the thermal damage by the rapid temperature change due to the difference in the coefficient of thermal expansion ($\alpha_{Al}=23 \times 10^{-6}$, $\alpha_{CF}=1.8 - 5.3 \times 10^{-6}$), the specimens were carefully heated up to the designed temperatures for 3.6 ks and cooled in the furnace after heat treatment. The specimens without heat treatment were used as standard specimens for comparison.

3. Apparatus for the measurement of AE signals

The 3-point bending test apparatus was designed to observe the AE signal produced during the application of load. The AE signals detected by a PZT-piezo electric sensor were transmitted to the AE-measuring device which is composed of an amplifier, filter, wave memory and micro-computer.

(1) 3-point bending test apparatus

The sample was placed on a pair of cylindrical supports of 3 mm diameter aligned parallel to each other with a spacing of 20 mm. The bending load was applied to the midpoint of the specimen by pressing with the side surface of a cylinder of the same diameter as that of the supports. Before testing, a weight and a tank of balanced weight filled with water were hung on both ends of a beam. The loading was performed by flowing the water from the tank. This loading method was employed to minimize the mechanical noises obstructive to the AE measurement. The load detected by a small-sized load cell was recorded by a pen-recorder.

(2) AE-detector

A PZT-piezo electric sensor 5 mm in diameter and 1 mm in thickness was glued directly on the top surface of the specimen. At the contact points between the specimen and the elements of the bending test apparatus, pieces of a teflon sheet 50 $\mu$m in thickness were inserted to reduce the noises produced from the local fracture by the stress concentration.

(3) AE measuring system

The memory capacity of the wave memory is 2 k words, and the sampling clock is 500 ns per word. The total gain of the amplifier is 40 dB, and the cut-off frequencies of the high-pass and low-pass filters are set at 100 kHz and 1 MHz, respectively. The filtered AE signal with the amplitude larger than the set trigger level is displayed on a cathode ray tube and transferred to the micro-computer to save in a floppy disk. The number of the words is 2048 for each transfer.

4. AE signal measurement

In the present experiment, the total number of AE counts (events) and the frequency spectrum of AE signals were investigated in correlation with the changes in the mechanical property and SEM fractography for various heat treatments.

(1) Total AE counts and AE counts for load level

The number of AE counts for various load levels and the total AE counts at various trigger levels were measured.

(2) The frequency spectrum analysis

For each testing, about 20 to 30 AE signals were sampled and saved in a floppy disk for spectrum analysis. The frequency spectrum is obtained by the following procedures.

(a) Nyquist frequency

The AE signal is analyzed by the fast Fourier transform (FFT). The sampling period is 500 ns and the sampling frequency is 2 MHz. The data sampling time and fundamental frequency are
The Nyquist frequency is
$$f_c = \frac{\text{sampling frequency}}{2} = 1 \text{ MHz},$$
then the cut-off frequency of the low-pass filter for antialiasing is 1 MHz.

**b) Removal of noise spectra**

As the internal noise of the amplifier may be superimposed on the AE signals, so in order to remove the frequency spectra of this noise, the blank test was performed 5 times both before and after testing, and an average of these 10 frequency spectrum data was substituted from that of the measured AE signal.

### III. Experimental Results and Discussion

1. **Maximum bending stress**

The maximum bending stress obtained from the fracture load is shown in Fig. 1 as a function of heating temperature. The figure shows that the specimen heated at 670 K retains the strength of 90 % of the non-heat treated, and the strength of the specimen at 790 K reduced to about 70%. The large degradation of strength takes place at temperatures between 670 and 790 K. The decrease of the strength is smaller at temperatures above 790 K. To be discussed in the following sections, the degradation will largely result from the change in the CF/Al-matrix bonding and partly from the formation of mixed layer of Al and carbon atoms previous to the disappearance of the CF/Al interface which can be seen in the SEM fractographs. Once this layer is formed, further inter-diffusion of both elements in this region will cause only small a change in the structure and in the fracture process, then the strength will not change so much at temperatures higher than 790 K.

2. **Fractography of the specimens**

After testing, the fractured surfaces were observed with a SEM. Figure 2(a) and (b)–(d) are those SEM fractographs of the non-heat treated specimen, and the heat treated ones at 670 K, 790 K and at 890 K for 3.6 ks, respectively. In the specimen without heat treatment, the fractured surface is very rough and pull-out of CF from Al matrix is obvious. The Al matrix shows ductile fracture surface as shown in Fig. 2(a). On the contrary, the fractured surfaces of the specimens heat treated at temperatures higher than 790 K tend to be more smooth and the number of pulled-out fibers decreases with increase of heating temperature. The specimen heated at 890 K clearly shows a simultaneous fracture of CF and the Al matrix. This suggests the brittle crack initiation due to the tight bonding of CF and Al by the formation of the compound, Al₄C₃, at the interface and the formation of the embrittled layer in the Al matrix adjacent to the fiber by the diffused carbon atoms from the fiber.

3. **Characteristics of AE signals**

1) **AE counts as a function of applied load**

The histograms in Fig. 3(a)–(c) show the change of AE counts by applied load at various threshold voltages of the wave memory. These figures show that in the specimens heated at higher temperatures, the AE signals can be detected from smaller load and specimens break at the earlier stage after the first AE signal was observed. For a larger threshold voltage, the specimen heated at 890 K emits AE signals at almost the final stage of the fracture.

2) **Total AE counts**

In Fig. 4 the total counts of AE counts are plotted against threshold voltages. In the non-heat treated specimen, AE counts for $V_t = 1 \text{ mV}$ is about 30% of that for $V_t = 0.3 \text{ mV}$.
Whereas, in the specimen heated at 890 K, the count for $V_t=1 \text{ mV}$ reduces to about 4% of that for 0.3 mV, and the amplitude of each AE signal is small. From the larger reduction of AE counts compared to the reduction of fracture load to about 70% of that of the non-heat treated one, two reasons considered are: the most part of elastic energy is retained until the specimen breaks, or the AE signals are miscounted because they are smaller than the sensitivity of the present measuring system. The former case is more likely, because the SEM fractography shows the simultaneous fracture of both components of the composite.

(3) Frequency spectrum analysis
The results of the FFT analysis of the AE signals are shown in Fig. 5 (a)-(c) for various load levels, $L/L_{\text{max}}$, where $L$ is the applied load and $L_{\text{max}}$ is fracture load.
(a) Non-heat treated specimen
A strong spectrum $a$ at 80 kHz can be seen for all specimens, so this component is not the specific characteristic of this specimen. The most typical spectra with two peaks, $c$ and $d$, and $e$ and $f$ are observed at 300–400 and 400–500 kHz, respectively.
(b) Heat treated at 670 K for 3.6 ks
A spectrum $b$ appeared at 120 kHz and its peak becomes larger with increase of load. The typical change in the spectrum diagram is that the spectrum $f$ becomes very strong compared to $e$. The peaks $c$ and $d$ are very small at the fracture, as is the case with that of non-heated specimen.
(c) Heat treatment at 790 K for 3.6 ks
Peak $b$ at 120 kHz becomes stronger especially at $L/L_{\text{max}}=0.9$, on the other hand, peaks $e$ and $d$ at 300–400 kHz become weaker and almost disappear at the rupture of the specimen. A new peak appears markedly at 480 kHz.
(d) Heat Treatment at 890 K for 3.6 ks
The peak heights of $e$ and $f$ become very

Fig. 2 SEM fractography of CFR-Al, non-heat treated (a), heated at 670 K (b), 790 K (c) and 890 K (d) for 3.6 ks, respectively.
small and c almost disappears. The peaks at about 120 kHz is more pronounced than the other specimens. The spectrum components can be considered to become simpler and this fact suggests that the energy release during the fracture process may be done by a much simpler fracture process in the absence of separation of CF from the Al matrix. This corresponds well to the change in SEM fractography.

As described above, the degradation of the strength of the composite by heating, due to the formation of the aluminum carbide at the interface of both components and its growth can be considered to be reflected on the AE signals.

The fracture process of the non-heat treated specimen may proceed as follows. The fracture
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( a ) $L / L_{\text{max}} = 0.73 - 0.81$

( b ) $L / L_{\text{max}} = 0.89 - 0.91$
of CF is followed by the separation of CF from Al by the propagation of splitting along their interface. This process will continue until the specimen breaks. A large amount of plastic deformation of Al is given once all the carbon fibers are broken.

If the specimen is heated at a temperature high enough to form aluminum carbide, the separation along the interface will be difficult to take place because of the formation of rigid bonding. On increasing the heating temperature, a considerable amount of the surface layer of CF is damaged by the formation of a compound and the embrittled region of Al by diffused carbon extends into the matrix. Under these conditions, CF and Al will break together to form a flat fractured surface with little plastic deformation. Thus the fracture mechanism tends to be simple and kinds of the corresponding AE signal source is decreased.

In the present experiment, the one to one correspondence between AE signal and each fracture process, such as the fracture of CF or the Al matrix, and the propagation of fiber/matrix splitting, as was obtained for CFRP, has not been clear yet; however, the correspondence of the AE spectra change to the change of the fractured surface is observed. In addition to this, the most important point is that the small change in the bonding at the interface of both components, which is not observable by SEM fractography, can be detected by the AE frequency spectrum.

IV. Summary

The correlation between the mechanical property change and the AE signals produced during the loading by the 3-point bend has been investigated in the carbon fiber reinforced aluminum composites. The results obtained are summarized as follows.

(1) In the specimens without heat treatment and those heated at a temperature lower than 670 K, the pull-out of CF and the ductile fracture of the Al matrix are observed by SEM fractography. The difference of the bonding between these specimens can be well recognized.
by the AE spectrum diagram, though the difference of the fractured surfaces can hardly be observed with SEM fractography. This suggests that the AE measurement will be a powerful tool for detecting the bonding conditions in FRM.

(2) In the specimens heat treated at higher temperatures, the interface between CF and Al disappears by inter-diffusion and the matrix becomes brittle, then the CF and Al fractures together to form a flat fractured surface. The AE signal with the reduced number of spectra in these specimens suggests that the fracture process tends to become simpler.

(3) The frequency of AE events depends on the microscopic fracture process of CF and Al. In the non-heat treated specimens large and small AE signals are detected randomly, while the specimens heated at higher temperatures emit smaller AE signals at lower applied loads and larger AE signals at higher loads. This suggests that the larger amount of the elastic energy is released at the final fracture stage in the latter case, in which the rupture of CF and Al must take place at almost the same time.

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REFERENCES

(1) A. A. Baker: Mat. Sci. and Eng., 17 (1975), 177.
(2) J. Morley: International Metal Review, Sept., (1976), 153.
(3) W. Kinner: Mat. Eng., 9 (1980), 60.
(4) S. Tai, D. Mahulikar, H. L. Marcus, I. C. Noyan and J. B. Cohen: Mat. Sci. Eng., 47 (1981), 145.
(5) K. Kobayashi and A. Kitamura: J. Japan Soc. Comp. Materials, 2 (1976), 60.
(6) S. Kohara and N. Mutoh: J. Japan Inst. Metals, 44 (1980), 271.
(7) S. Kohara, N. Mutoh and Y. Imanishi: ibid., 43 (1979), 189.
(8) I. H. Khan: Met. Trans. A. 7A (1976), 1281.
(9) S. J. Baker and W. A. Bonfield: J. Mat. Sci., 13 (1978), 1329.
(10) H. Ohtsuka: J. Japan Soc. Comp. Materials, 10 (1984), 102.
(11) E. Nakata, Y. Kagawa and H. Terao: ibid., 9 (1983), 115.
(12) K. Shimomura, H. Suzuki and H. Sekine: ibid., 11 (1985), 21.
(13) S. Umekawa, K. Wakashima, S. Yoda, R. Takahashi and I. Ioka: Composite Materials: Mechanics, Mechanical Properties and Fabrication, Ed. S. Umekawa and T. Akasaka, Japan Society for Composite Materials, Tokyo (1981), p. 245.
(14) M. Suzuki, H. Nakanishi, M. Iwamoto, E. Jinen, Z. Maekawa and A. Mori: Composite '86; Recent Advances in Japan and the United States, Japan Society for Composite Materials, Tokyo, (1986), p. 631.
(15) C. Johnson, K. Ono and D. Chellman: ibid., p. 647.