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The effect of heat treatment on Young's modulus, damping, and microhardness of SiC/Ti–15–3

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Mechanical and physical properties of a laminated, continuous SiC-fibre-reinforced Ti–15V–3Cr–3Al–3Sn (Ti–15–3) composite were analysed. The dynamic elastic modulus and damping of specimens with both unidirectional and cross-ply laminates were measured with the PUCOT (piezoelectric ultrasonic composite oscillator technique). The values of density, elastic modulus, and damping were evaluated with respect to heat treatment of the specimens. Metallographic examination was also done on the specimens to assess any changes in the microstructure in terms of changes in the mechanical or physical properties.

1. Introduction

Metal matrix composites (MMCs) offer greater strength and stiffness than matrices provided by polymers. They are also able to perform well at elevated temperatures and provide greater fracture toughness, relative to polymers. Because of their excellent strength-to-density and stiffness-to-density ratios, there are many potential applications for MMCs. These include uses in engines and hypersonic flight vehicles.

Investigations have been done in tensile deformation with respect to heat treatment [1], microstructural and property changes as a function of ageing [2], and initial microstructure and macrostructure of the SiC/Ti–15–3 material [3]. These studies have shown that the properties of SiC/Ti–15–3 can change drastically with heat treatment processes. It has been determined that the properties of the composite can vary significantly from those of the monolithic matrix.

The purpose of this study was to determine how the mechanical properties, such as elastic modulus and damping, of the SiC-fibre-reinforced Ti–15–3 changed after heat treatment. Metallography was also done on the specimens in an attempt to understand the reasons for these changes.

2. Experimental Procedure

2.1. Materials Tested

The composite material was made by alternating layers of Ti–15V–3Cr–3Al–3Sn foils and continuous SiC SCS-6 fibres. The unidirectional and cross-plied fibre layups were hot isostatically pressed at high temperatures using proprietary consolidation procedures. Eight rows of fibres were used, resulting in a total composite thickness of approximately 2 mm. The fibre volume fraction was nominally 34% [1]. Specimens with 15% and 41% fibre volume were also manufactured and tested.

Nineteen specimens in all were tested, with varying heat treatments. Sixteen of the specimens were composite specimens with varying directions for the laminate, while three monolithic specimens were tested.

The specimens were subjected to two different heat treatments: 700 °C/24 h and 700 °C/24 h + 427 °C/24 h. Each different type of specimen was evaluated as subsequent heat treatments were added.

2.2. PUCOT measurements

The PUCOT (piezoelectric ultrasonic composite oscillator technique) was used to determine dynamic Young's modulus, mechanical damping, and strain amplitude for the specimens. The test incorporated two identical alpha-quartz piezoelectric crystals (a drive and a gauge), and the test specimen. The components were bonded together using a high strength glue. The piezoelectric drive crystal excited longitudinal ultrasonic stress waves in the test specimen, which was cut to the appropriate resonant length. The gauge crystal then responded to the stress waves excited in the specimen. Signals into and out of the system were in the form of alternating voltages. Data compilation was computerized for ease in calculation and flexibility of outputs. The outputs gained from these experiments were Young's modulus (E), strain amplitude, and damping (Q^{-1}) [4]. Damping is a measure of the dissipated vibrational energy divided by the total

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stored elastic energy per cycle. More detailed descriptions of the PUCOT have been given elsewhere [5–7].

These experiments were conducted at room temperature with 80 kHz quartz crystals. Specimens were cut to their appropriate resonant lengths using a diamond cutting machine.

The PUCOT test is a favourable alternative to tensile testing for finding Young's modulus of a specimen. Since the test is non-destructive, specimens can be used for repeated testing or for other experiments.

2.3. Metallography

The purpose of the metallography on the specimens was to find differences in microstructure due to the heat treatments. Three unidirectionally reinforced and three monolithic Ti–15–3 specimens were mounted. The mounting material was black epoxy (bakelite). The following sequence of sand paper grit sizes and polishing compounds were used: 240, 320, 400, 600 and 0.25 μm diamond paste, and 0.05 μm alumina powder in distilled water. The unidirectionally reinforced specimens were not etched, while the monolithic specimens were etched with a solution of 2 ml HF and 3 ml HNO_3 added to 100 ml of water at room temperature. Colour photographs were taken of the composite specimens with a polarizer to attempt to bring out the differences in the two materials. The matrix-only photographs were taken in black and white.

2.4. Microhardness measurements

Microhardness measurements were made on the three 0° composite specimens using a Buehler Micromet II Digital Microhardness Tester. A load of 2.94 N was used for 15 s. Indentations were made on both the SiC fibres and the matrix.

3. Results

First, density measurements were made using Archimedes' method for all of the specimens since these values were needed for the PUCOT measurements. These results can be found in Table I. Next, the modulus and damping were found using the PUCOT. These results can be found in Tables II and III, respectively. The results for the microhardness testing can be found in Table IV.

4. Discussion

In Figs 1 and 2 the trends of modulus for the matrix and the 0° specimens with successive heat treatments can be seen. The reference was the Young's modulus of the as-received specimen.

Fig. 1 indicates that the Young's modulus for all specimens, regardless of percent fibre, increased with increasing ageing. The matrix showed an 8.5% increase in modulus with the original 700 °C heat treatment, and a 24.1% increase with the 700 °C and the 427 °C heat treatments. Fig. 2 indicates that the composite specimens showed increasing trends in modulus

TABLE I Densities of specimens in $kg\,m^{-3}$

	As received	700 °C/24 h	700 °C/24 h + 427 °C/24 h
Matrix	4747	4987	4763
[0]8	4139	4097	4079
[0]8 (15v/o)	[a]	4499	4491
[0]8 (41v/o)	[a]	4152	[a]
[90]8	[a]	4094	4097
[90/0]2s	[a]	4149	4064
[0/90]2s	[a]	4152	4131
[± 45]2s	[a]	4139	4033
[± 60]2s	[a]	4151	4137

[a] No specimen.

TABLE II Young's modulus of specimens in GPa

	As received	700 °C/24 h	700 °C/24 h + 427 °C/24 h
Matrix	87.4	94.8	108.5
[0]8	200.5	205	209.9
[0]8 (15v/o)	130[a]	137.8	153.3
[0]8 (41v/o)	210[a]	232.4	[b]
[90]8	111[a]	132.6	146.3
[90/0]2s	128[a]	157.1	165.3
[0/90]2s	128[a]	155.1	165.3
[± 45]2s	107[a]	122.7	134.9
[± 60]2s	104[a]	121.7	139.4

[a] Values from tensile tests, NASA Lewis Research Center [8].
[b] No specimen.

TABLE III Damping $\times 10^{-4}$ (Q^{-1})

	As received	700 °C/24 h	700 °C/24 h + 427 °C/24 h
Matrix	2.7	1.3	0.7
[0]8	5	2.8	0.3
[0]8 (15v/o)	[a]	3.7	0.6
[0]8 (41v/o)	[a]	2	[a]
[90]8	[a]	3.2	6.1
[90/0]2s	[a]	12.7	19.5
[0/90]2s	[a]	5.1	19.3
[± 45]2s	[a]	6.8	7.4
[± 60]2s	[a]	9.1	11.9

[a] No specimen.

TABLE IV Microhardness: mean and standard deviation (H_v)

	Matrix	SiC
As received	221.6 ± 10.5	1669 ± 145
700 °C/24 h	242.4 ± 9.9	1896 ± 186
700 °C/24 h + 427 °C/24 h	327.1 ± 11.0	1786 ± 156

as the percent volume of SiC increased for the three different heat treatments, as one would expect from a micromechanics analysis:

$$E_{comp} = E_{matrix}(1 - V_{fibre}) + E_{fibre}V_{fibre}$$

Fig. 3 indicates that the mechanical damping decreases with additional heat treating. Both the matrix and 0° composite specimens had a decrease in

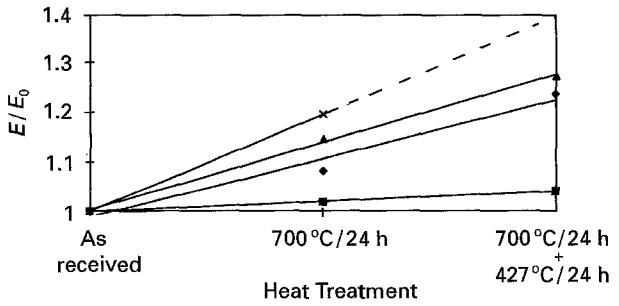

Figure 1 Young's modulus as a function of heat treatment for SiC/Ti-15–3. ◆ matrix; ■ [0]8 ▲ [0]8 (15 v/o); × [0]8 (41 v/o).

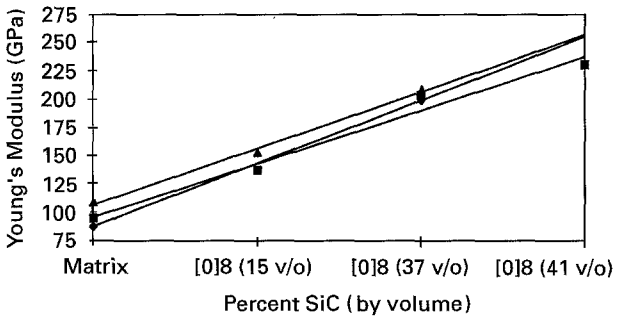

Figure 2 Young's modulus as a function of percent SiC (by volume) for SiC/Ti–15–3. ◆ as received; ■ 700 °C/24 h; ▲700 °C/24 h + 427 °C/24 h.

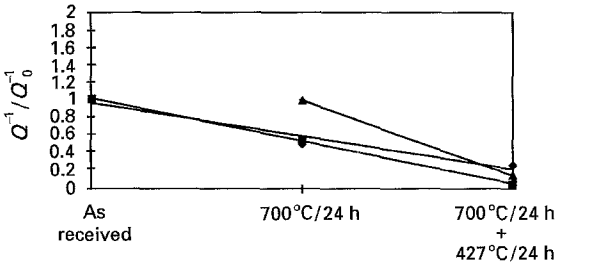

Figure 3 Mechanical damping as a function of heat treatment for SiC/Ti–15–3. ◆ matrix; ■ [0]8; ▲ [0]8 (15 v/o).

damping as the various heat treatments were added. This decrease in damping with an increase in modulus agrees with general observations on a variety of materials [9]. However, the other laminate specimens showed an increase in damping as the heat treatment was applied. The reason for such behaviour is not yet understood.

The metallographic results for the unetched 0° composite specimens showed little differences between the specimens that had been heat-treated and those that had not. The micrographs of these specimens can be found in Figs 4–6. The layered microstructure of the SiC fibre can be seen in the photographs. This includes the carbon core, a pyrolytic coating, the actual SiC region, the carbon-rich coating, and the reaction zone [3]. There were no observable changes in the thickness of the reaction zone between the fibre and the matrix for either of the heat treatment conditions. The coloured micrographs revealed no additional information.

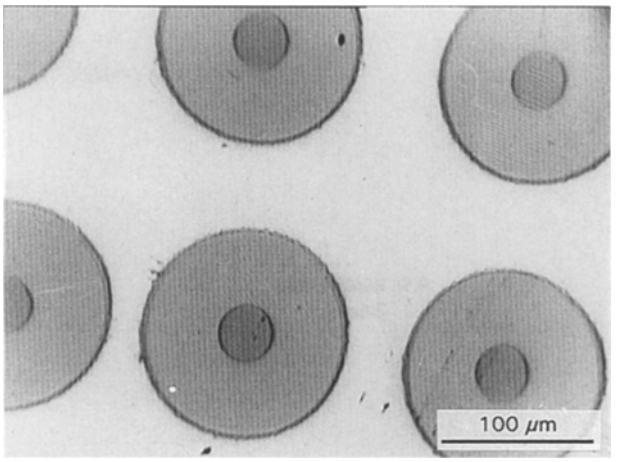

Figure 4 [0]8 as received specimen. Actual fibre diameter 0.14 mm.

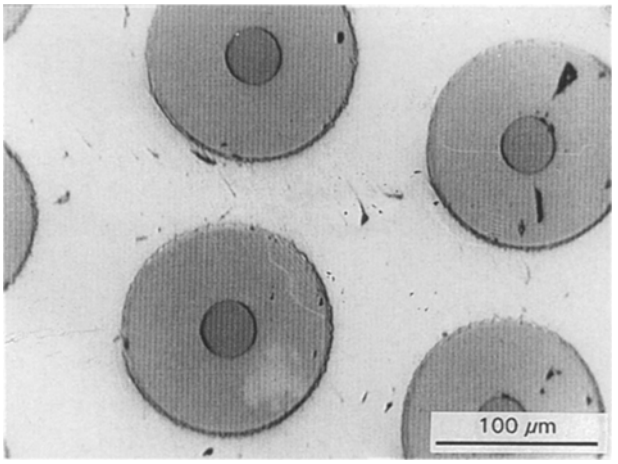

Figure 5 [0]8 700 °C/24 h specimen. Actual fibre diameter 0.14 mm.

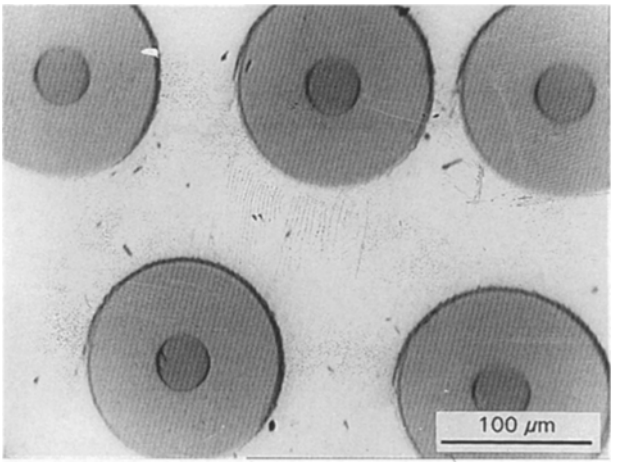

Figure 6 [0]8 700 °C/24 h + 427 °C/24 h specimen. Actual fibre diameter 0.14 mm.

The three monolithic specimens that were examined metallographically were the as-received specimen, the specimen that had received the 700 °C heat treatment, and the specimen with both the 700 °C and the 427 °C heat treatments. These can be seen in Figs 7–9. Fig. 7 shows the as-received microstructure, which contained many precipitates of a beta-prime phase, which is a predecessor to the alpha phase [2]. After the

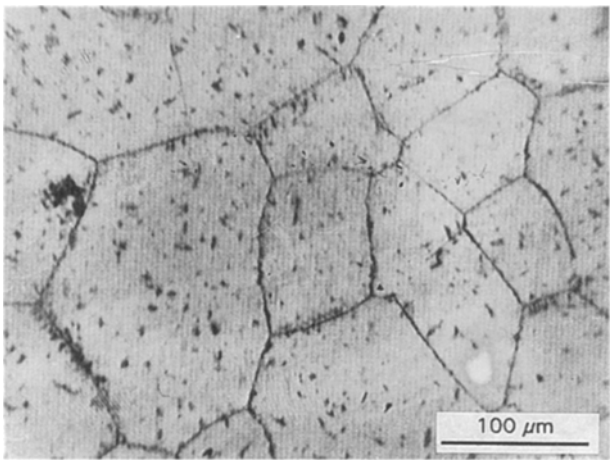

Figure 7 Matrix as received specimen. ASTM grain size number = 5.

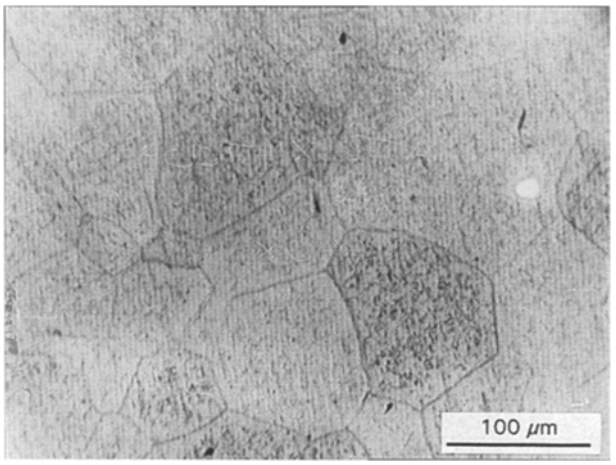

Figure 8 Matrix 700 °C/24 h specimen. ASTM grain size number = 5.

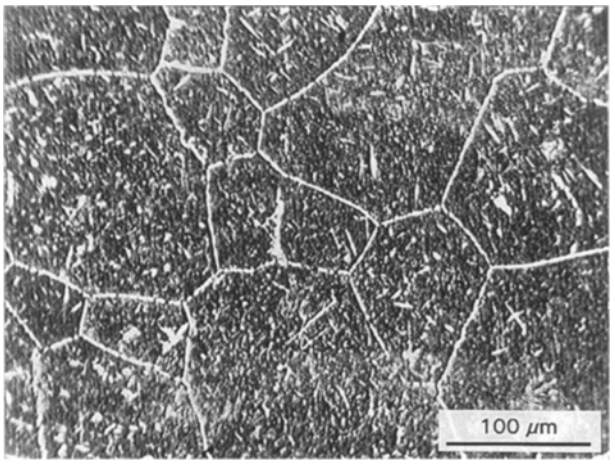

Figure 9 Matrix 700 °C/24 h + 427 °C/24 h specimen. ASTM grain size number = 5.

ageing of the matrix at 700 °C for 24 h, the dark beta-prime phase was replaced by large needles of alpha-Ti (Fig. 8), which are more evident at the grain boundaries. This is the primary hardening phase of the matrix [2]. In Fig. 9 the same alpha-Ti needles can be seen, together with a high volume fraction of finer low temperature ageing alpha-Ti precipitates [10].

The effects of heat treatment on the hardness of the unidirectionally reinforced specimens can be seen in Fig. 10. Each data point represents an average of at least five measurements for each specimen, using Vickers hardness numbers (H_v). The range of the hardness values per specimen was approximately $\pm$ 10 H_v for the matrix, and $\pm$ 160 H_v for the SiC fibres. These tests were measured on the actual SiC part of the fibre, and not the carbon core. It can be seen that while the SiC fibres are much harder than the matrix, the heat treatment affected the matrix to a greater extent; the matrix showed almost a 50% increase in hardness with both heat treatments, while the hardness of the SiC fibres remained within 10% of its original hardness.

Similarities can be noted between the increase in modulus with heat treatment and the increase in hardness with the same heat treatment. The changes in the matrix properties, with modulus and with hardness, seem to dominate the changes in composite properties. These trends of an increase in elastic modulus with an increase in hardness reflect the general trends that are documented for many materials as bar charts in the book by Ashby and Jones [11].

Granato–Luecke (G–L) analyses were also performed on the two [$\pm$ 45]2*s* specimens to assess the strain amplitude dependence on damping [4]. These two specimens were the only ones that showed this trend of increased damping as the strain amplitude was increased. The Granato–Luecke analysis provides a value for dislocation density that may later be compared to one measured by electron metallographic techniques [4]. For the specimen with the 700 °C heat treatment, the G–L analysis yielded a mobile dislocation density of 2×10^4 cm^{-2}. The specimen with both heat treatments also had a mobile dislocation density of 2×10^4 cm^{-2}. For these calculations, a lattice parameter of 3.23×10^{-10} m, a dislocation network length of 3.5×10^{-4} m, and a Burger's vector of 2×10^{-10} m were assumed for the matrix. The values of mobile dislocation density are relatively low, but experimental values determined by transmission electron microscopy are not available.

It is interesting to note that these [$\pm$ 45]2*s* specimens are the only ones that showed this strain amplitude dependence on damping. This could be caused by the possibility that these [$\pm$ 45]2*s* specimens experience some slipping of the fibre/matrix interface when subjected to higher strains.

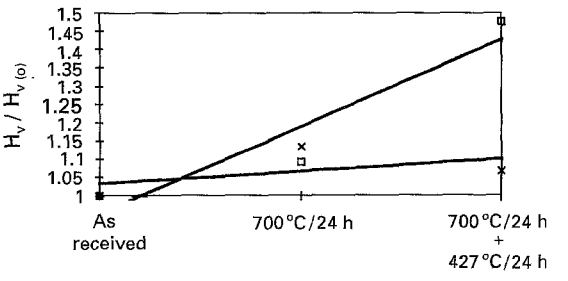

Figure 10 Microhardness as a function of heat treatment for SiC/Ti–15–3. □ matrix; × SiC.

5. Conclusions

Heat treatment affected the properties of dynamic modulus, damping and hardness in the SiC/Ti–15–3 specimens. The specimens showed a definite increase in modulus as heat treatment was added. Though all of the different laminate specimens showed different rates of increase, a trend can be determined, especially in the matrix and the 0° composite specimens. The damping values also showed a pattern. The 0° and matrix specimens showed decreased damping, while all of the other laminate specimens showed an increase as heat treatment was applied. It is yet to be determined if this trend is random or if there is some significance to the fact that some of the specimens had an increase in damping while others experienced a decrease. From the results of the metallography and microhardness testing, it can be concluded that the phase changes in the matrix lead to the changes in the mechanical properties of modulus and microhardness.

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