Research report

Initial-stage hot-pressing of SiC fibre/Ti monotapes

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The initial compaction under uniaxial hot-pressing conditions of titanium monotapes containing silicon carbide fibres is studied for variable matrix/fibre volume ratio, temperature (800–950°C) and compaction stress (10–40 MPa). The matrix compaction rate is predominantly affected by the latter two variables, less by temperature. The sample pore structure is strongly influenced by the preform packing characteristics including fibre-to-matrix volume ratio and irregularities in fibre arrangements.

Key words: *SiC/Ti monotapes; hot-pressing; porosity; preform packing*

Due to the high reactivity and high melting point of titanium, composites of fibre-reinforced titanium and its alloys are produced by solid-state consolidation techniques such as diffusion bonding, hot-pressing, hot or cold hydrostatic pressing or high strain rate compacting. The densification kinetics of titanium-based powders[1–3] and foils[4,5] have been investigated and modelled by several authors, but are difficult to apply with metal-matrix composites because of the increased geometrical complexity that results from the presence of rigid fibres or particles. We present here some of these complicating effects, together with an exploratory examination of the influence of temperature, compaction stress and relative amount of matrix and fibres on the consolidation kinetics by hot-pressing of continuous silicon carbide fibre/titanium monotapes.

EXPERIMENTAL PROCEDURES

Fibres used were continuous silicon carbide monofilaments of two types: 142 μm diameter SCS-6™ monofilaments (from Textron, Lowell, MA, USA) and 90 μm diameter Sigma™ monofilaments (from British Petroleum, Farnborough, Hants, UK). Aligned fibres were plasma-sprayed with unalloyed titanium at Matra (Vélizy, France), to form a monotape having a total thickness of around 200 μm. The matrix contained 0.34% O, 0.003% N, 0.004% H, 0.04% Fe, 0.002% Zr, balance Ti. Two types of monotape were fabricated: fibre-rich monotapes with a volume fraction of fibres between 0.53 and 0.58 (Fig. 1), and matrix-rich monotapes with a volume fraction of fibres between 0.39 and 0.46.

The as-sprayed material was cut into square pieces 10 mm wide, by sandwiching the monotape between cardboard sheets and sectioning with a sharp blade. During cutting, fibre-rich monotapes tended to split lengthwise

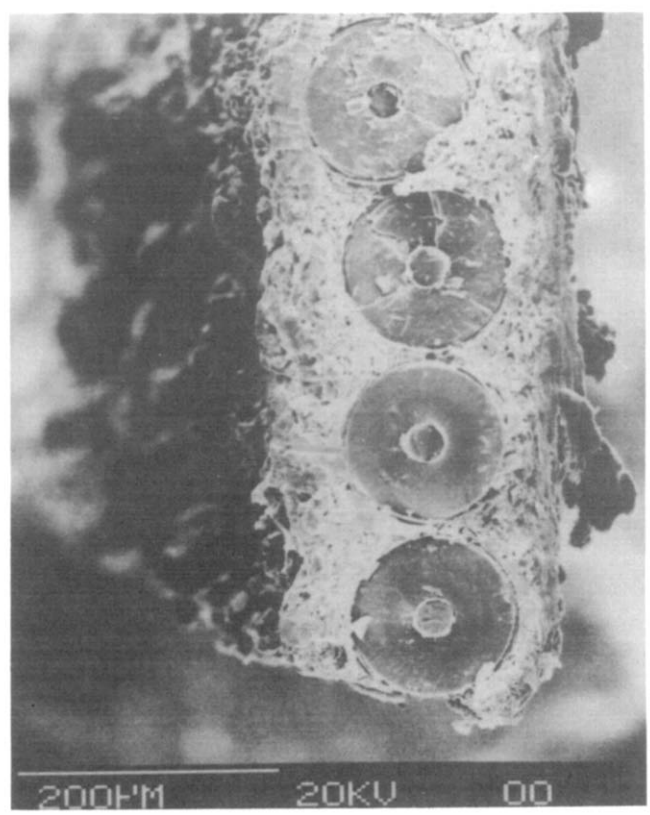

Fig. 1 Scanning electron micrograph of as-recieved fibre-rich SCS-6™ monotape

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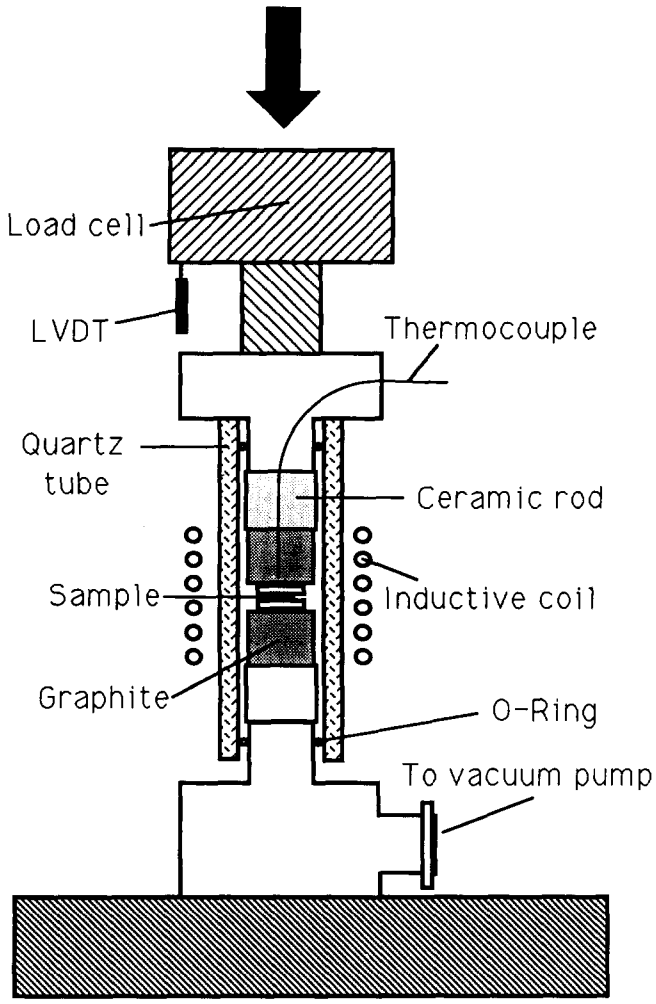

Fig. 2 Schematic of hot-pressing apparatus

between the fibres to form monotape pieces containing from two to about 50 fibres. One gram of cut monotape material was then placed within a molybdenum die protected by stainless steel shim on all sides, by carefully aligning the monotape pieces parallel to one another. Titanium sheets 75 μm in thickness were placed below and above the packed preform. Graphite and a molybdenum piston, separated with boron-nitride-coated foils of stainless steel, were then stacked on top of the preform. The loaded die was finally placed between two graphite susceptors held between two columns of insulating ceramic spacers, enclosed within a quartz tube around which an induction coil was placed. The temperature of the specimen was measured by a thermocouple in contact with the top of the molybdenum piston. A schematic of the experimental set-up is given in Fig. 2.

The quartz tube was evacuated to a pressure below 10^{-3} Pa and the specimen was outgassed at 300°C for about 30 min under an applied stress of 1.6 MPa. It was verified by retrieving samples at this stage that no consolidation of the monotapes took place during outgassing. The specimen was then heated to the consolidation temperature at a rate of about 3°C s^{-1} and held at temperature for ~5 min in order to stabilize the temperature. Stress was then applied to the specimen by the crosshead of the mechanical testing machine, travelling at a speed of 2.1×10^{-4} m s^{-1}, while its position was measured by a linear variable displacement transducer having an accuracy of ±2 μm. Crosshead motion was stopped when the load measured by the load cell corresponded to 110% of the nominal consolidation stress. The measured load then decayed due to consolidation of the specimen. When the stress reached 90% of its nominal value, motion of the crosshead was started again, and the same procedure was repeated. All experiments were performed under a residual gas pressure below 10^{-2} Pa, measured at the entrance of the vacuum pump. The temperature oscillated around the nominal value by ±15°C. Ensuing thermal expansion of the compression column introduced periodic oscillations in load value that were smaller than 10% of the nominal load value. After 900 s of consolidation, the applied pressure was rapidly reduced to 1.6 MPa and the specimen cooled at about 0.5°C s^{-1} to 300°C.

The average final height of the hot-pressed specimen was measured, and the specimen was cut with a low-speed diamond saw, cold mounted in epoxy, ground on silicon carbide paper and polished with diamond slurries. The volume fraction of pores was then determined by point-counting, and that of fibres by counting the fibres, on cross-sections perpendicular to the fibres.

RESULTS

Kinetics of hot-pressing

The volume fraction of pores $V_p(t)$ of the specimen as a function of time was back-calculated from the crosshead position measured at the end of each period of crosshead movement, knowing the final specimen height and pore volume fraction. The initial porosity, back-calculated to the first loading cycle, varied, being as high as 40 vol%.

The final sample porosity was very sensitive to the initial packing of monotapes: under the same conditions of stress, temperature and time, specimens with a few large monotapes reached much higher final densities than those with many smaller monotape pieces. Scatter in the data induced by fibre packing effects was too large to allow comparison with densification models for bulk titanium[6] or monotapes[7], or to detect variations in the rate of compaction across the transus temperature. To compare overall consolidation rates despite significant scatter in the data, we define a time t^* at which a compaction rate of 5×10^{-5} s^{-1} is reached. This compaction rate was chosen because it is (1) sufficiently low not to predominantly reflect densification by rearrangement, and (2) sufficiently high to be accurately measurable. Comparison of t^* from one sample to another then allows for a rough comparison of the overall rate of matrix densification for different process parameters, despite significant differences in the final porosity of the samples. t^* is plotted in Fig. 3 as a function of the applied stress for matrix-rich monotapes containing Sigma™ fibres. It is apparent that, within practically relevant process parameter ranges, the consolidation temperature exerts a much smaller influence on t^* than the applied stress. Also plotted in Fig. 3 are data for fibre-rich monotapes, which are systematically shifted to smaller t^* values (most likely because t^* corresponds to a higher intrinsic rate of densification in the matrix of these composites). These monotapes reflect the same trend for the effect of pressure

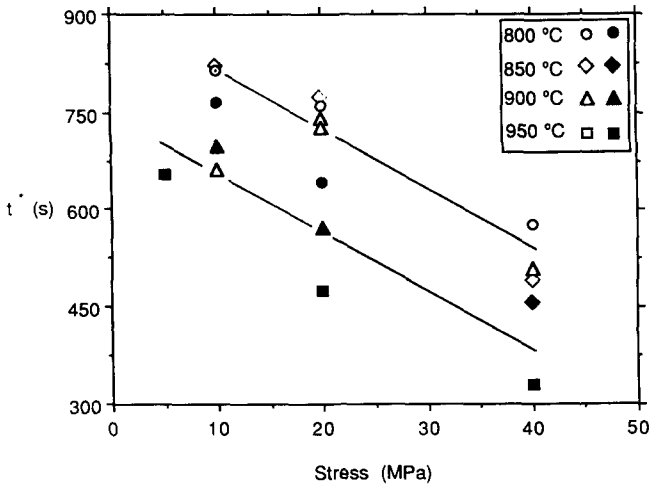

Fig. 3 Time t^* as a function of applied stress and temperature for matrix-rich specimens (open symbols) and fibre-rich specimens (filled symbols) of Sigma™ fibre-reinforced titanium monotapes

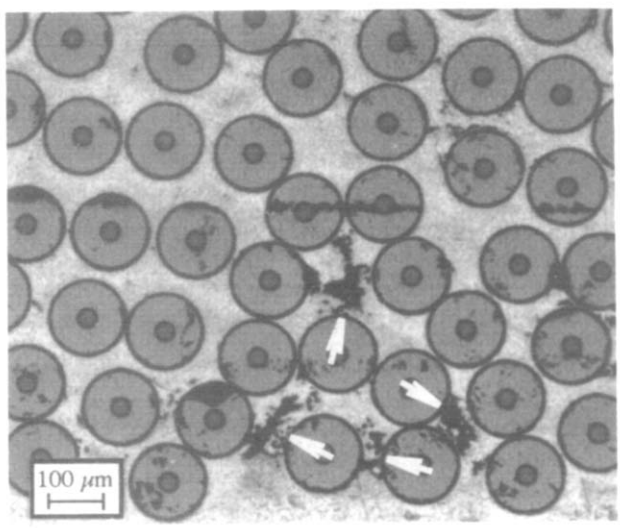

Fig. 4 Optical micrograph of a compacted sample showing a stack of touching fibres forming an arch shielding a matrix region with pores (arrows), while the surrounding unshielded matrix is virtually pore-free (SCS-6™ fibre, 900°C, 40 MPa). The external load was applied in the vertical direction of the micrograph

and temperature, which was also found in SCS-6™ fibre-reinforced monotapes (albeit with more scatter in the data, most likely because only fibre-rich material was available).

Microstructural effects due to initial packing of monotapes

Two microstructural effects which influenced significantly the final degree of consolidation were frequently observed in the present experiments:

1) *matrix shielding by contacting fibres.* In certain cases, matrix flows from the region between two neighbouring fibres to fill adjacent porosity. This results in contacting fibres which bear the load applied during the compaction operation and may locally shield a matrix region from the applied stress. Fig. 4 shows the effect of a stack of contacting fibres creating a vault over a region of high porosity in a region where the surrounding matrix is pore-free. This effect, similar to one observed by Lange *et al.*[8] with spherical inclusions, is to be expected when the starting porosity is high and/or the amount of matrix low. A similar effect arises when non-parallel fibres extrude matrix material that separates them and touch at isolated points.
2) *monotape end-effects.* When a monotape ends between two wider monotapes, a triangular cavity generally results at the end of the narrower monotape, Fig. 5. These pores were found to largely determine the final average porosity of specimens with mostly wide monotapes. Such defects, mostly created when cutting the monotapes to fit the die dimensions, may also arise when well-stacked monotapes break at weakest interfibre links during macroscopic deformation to fit a shaped die upon consolidation.

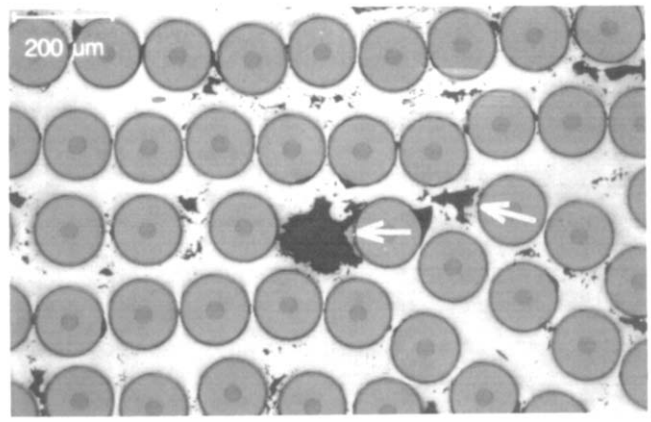

Fig. 5 Optical micrograph showing porosity (arrow) resulting from narrow monotapes sandwiched between broader monotapes (SCS-6™ fibre, 850°C, 20 MPa)

DISCUSSION AND CONCLUSION

Several investigations of the reactivity of SiC monofilaments in unalloyed titanium have shown that there is a significant reaction layer at the fibre/matrix interface[9,10]: in the temperature interval 800–950°C, for a given reaction time, the reaction layer thickness increases by a factor of 2.8 to 5.5. The present experiments, in which the compaction stress was found to exert a stronger effect on the compaction rate than temperature, indicate that within practical ranges, there is little incentive to raise the temperature to increase the compaction rate. Since the maximum compaction stress is limited by mechanical damage to the fibres, the initial fibre and matrix packing configuration emerges as the most important factor governing both the densification rate and the final porosity of the composite. This conclusion is reinforced by the strong influence on consolidation kinetics exerted by various defects in fibre packing, which were exacerbated in these experiments due to the small size of the samples.

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