Toughness of metal fibre/ceramic matrix composites (MFCs) after severe heat treatments

S. K. Lam and T. W. Clyne*

This paper concerns the effect of heat treatment on the toughness of metal fibre reinforced ceramic composite. Drawn fibres of 304 and 310 stainless steel have been used. Their response to heat treatment in air has been investigated, in terms of both oxidation kinetics and tensile properties. This has been correlated with measured fracture energies of composite containing these fibres, after similar heat treatments. As received, 304 fibres exhibit greater ductility and work of deformation than 310 fibres. However, severe heat treatments (up to 1170°C) cause 304 fibres to become oxidised and embrittled, whereas 310 fibres retain good strength and ductility. Composites subjected to these heat treatments become embrittled when they contain 304 fibres, but retain their toughness when reinforced with 310 fibres. These data are consistent with predictions from a previously published model of the fracture process, based on energy absorption being dominated by fibre pullout and/or fibre plasticity.

Keywords: Ceramic matrix composites, Short fibre composites, Fracture toughness, Fibre/matrix bond, Modelling

Introduction

This study is oriented towards the 'Fiberstone' family of composites, containing stainless steel fibres in a (slurry cast) matrix predominantly composed of alumina and aluminosilicate phases. Recent work[1] has highlighted the importance of the tensile properties of the fibres in conferring toughness on these composites. Since these composites are commonly used at high temperatures, and since their toughness is of central importance, there is considerable interest in how such fibres retain these properties (notably tensile strength and ductility) after prolonged heating.

Oxidation resistance in stainless steels is mainly achieved via passivation in the form of a surface layer of Cr_2O_3, which is highly stoichiometric and resistant to the passage of electrons and ions. It does thicken on heating, perhaps eventually consuming all of the available Cr and leading to the formation of other oxides (such as iron oxides), but this process is often slow, even at relatively high temperatures. The minimum Cr content of the steel required to form such a protective layer is usually taken to be ~13 wt%, although many stainless steels have much higher Cr levels than this. Austenitic stainless steels, such as 304 and 310, also contain significant levels of nickel, which stabilises the austenitic phase. As a general rule, steels with higher Cr contents have better resistance to high temperature oxidation. For example, the specification of 310 is typically for a Cr content ~24–26 wt%, whereas that for 304 is ~18–20 wt%, and the former is known to exhibit better oxidation resistance than the latter. It may also be noted, however, that Al is sometimes added to stainless steels, with the specific objective of creating an Al_2O_3 protective layer having superior stability to Cr_2O_3 at very high temperatures.[2,3] Furthermore, as shown by Hsu and Tsai,[4] the introduction of silicon into the surface layers (of 310) can enhance its oxidation resistance. The mechanisms responsible for the oxidation characteristics of austenitic stainless steels are in general fairly well understood.[5–7]

There have, of course, been many studies of how oxidation and degradation of mechanical properties of stainless steels proceed during heat treatments, but only a few such investigations[2,3,8–10] have been focused on the very high temperatures (above ~800–900°C) of prime interest here. This is mainly because these steels would not normally be used at such temperatures, since, as with virtually all steels, they would soften to such an extent that their use would be very problematic. Of course, the situation with Fiberstone composites is different, since the matrix remains relatively unaffected by heating to this temperature range, and in practice, these composites are being used in mechanically demanding applications requiring prolonged exposure to temperatures >1000°C.

There has been very little previous work concerning the effect of heat treatment on ceramic matrix composites reinforced with metal fibres. Faiyadh and Al-Ausi[11] reported that the tensile strength of concrete reinforced with fibres was higher than for unreinforced concrete, both before and after heat treatment (at up to 800°C),

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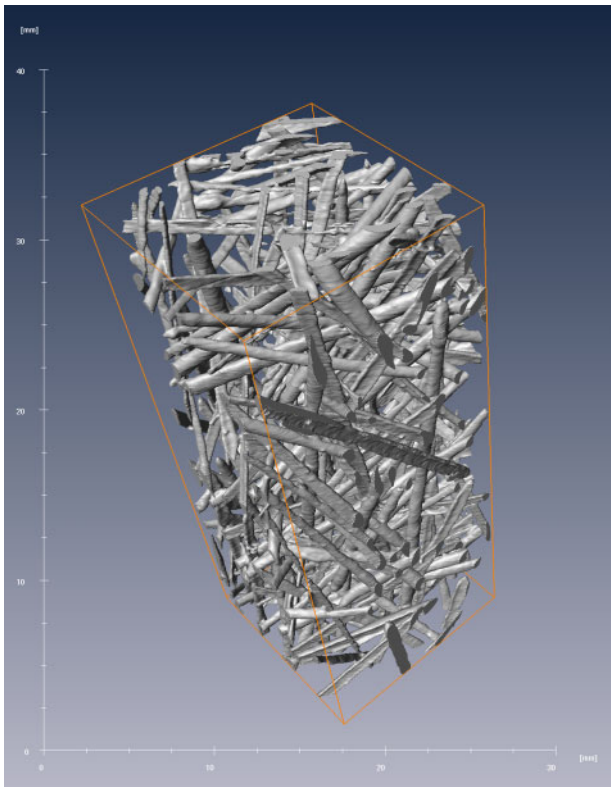


1 Tomographic data obtained from composite, showing reconstructed perspective view of fibre architecture; vertical direction is vertical axis of casting operation

with steel fibre reinforcement giving higher strengths than when glass fibres were used. Purkiss[12] measured compressive and flexural strengths of steel fibre reinforced concrete at up to 800°C, reporting that the reinforcement had beneficial effects (although the fibre contents ranged up to only 1·5 vol%). There appear to have been no previous studies concerning the effect of heat treatment on the toughness of metal fibre reinforced ceramic composites.

In the present paper, attention is concentrated on the oxidation resistance and retention of mechanical properties exhibited by fibres of 304 and 310 after a range of heat treatments. This relates specifically to these fibres, with particular diameters and surface characteristics, as well as specific compositions, rather than to 304 and 310 steels more generally. These results are correlated with the toughness of the corresponding composites, subjected to heat treatments after production.

Experimental

Material and specimen production

Fibre production

The fibres, which were supplied by Hunan Sunshine Steel Fiber Co. Ltd, China, were produced by cold drawing, with a final annealing treatment (sufficient to cause recrystallisation). They were 500 μm in diameter and 25 mm long (including 3 mm sections at both ends, which had been given a hooked shape). It may be noted that these fibres are uniformly cylindrical in section, in contrast to melt extracted fibres, which were used in the previous study.[1] The compositions of the drawn fibres were nominally those of 304 and 310 specifications. The compositions were verified by energy dispersive X-ray analysis of polished sections in a JEOL 5800LV scanning electron microscope. The outcome of this operation is shown in Table 1. The values are all within the corresponding specification ranges. It may be noted that the 310 contains ~3%Si, which is known[4] to enhance oxidation resistance.

Table 1 Measured compositions of fibres

	Composition/wt-%					
Type	Cr	Ni	Si	Mn	Al	Fe
304	18·3	8·5	0·7	0·0	0·0	Balance
310	25·2	17·9	3·1	1·4	0·0	Balance

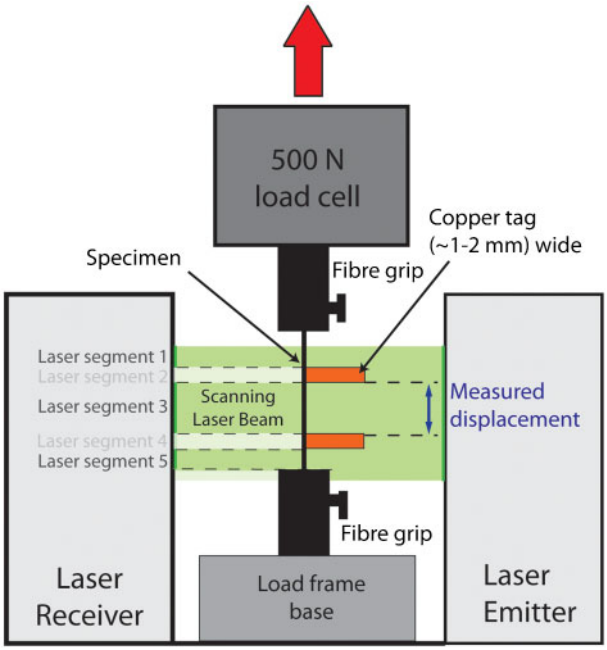
Composite production by slurry casting

The composite samples used in the present study are effectively part of a family of materials marketed under the trade name of 'Fiberstone', which are normally supplied in the form of finished ('cast') components, rather than as stock material. The specimen size in the current work was a square section rod, produced (at Fibretech Ltd) by packing fibres into a wooden mould and pouring a slurry into the cavity. Further details of the procedures employed, and the nature of the matrix, are provided elsewhere.[1] An indication of the fibre orientation distribution resulting from this type of processing is given by the X-ray tomography visualisation shown in Fig. 1, where it can be seen that, at least to a first approximation, the distribution is isotropic. Details of the methodology employed to produce this image are given elsewhere.[1] (The image shown is actually from a specimen containing melt extracted fibres, but the orientation distributions tend to be similar for the two types of fibre.) There have been previous studies[13] of such distributions in metal fibre reinforced ceramic composites (MFCs), but of course the outcome is highly specific to the processing route concerned. In fact, even for the process employed here, it should be recognised that the result depends on exactly how the fibre packing is carried out and, indeed, there is scope for the intentional creation of preferential fibre alignment.

Fibre oxidation

Oxidation testing was carried out in air at 1000 and 1170°C for periods of 5, 15, 35 and 85 h, with the fibres contained in quartz ampoules sealed at one end, the other end having been drawn down to a relatively small, open neck, in order to minimise the danger of material being lost during heat treatment and weighing. Spallation of oxide did tend to occur in some cases, but these spalled fragments remained within the ampoules. The ampoules weighed ~30 g, and the initial weight of a bundle of fibres within an ampoule was ~2 g. Weighing was carried out using a balance with a resolution of ~10 μg. Ampoules were removed from the furnace for each weighing operation, and only took ~30 min to cool to room temperature.

Interest in the oxidation behaviour of these fibres is oriented specifically towards the mechanical performance of the composite after heat treatment. Of course, the presence of the matrix is likely to affect oxidation of the fibres, although in practice it is well established that the matrix of Fiberstone composites has a relatively high level (~10–15%) of interconnected porosity and the oxidation behaviour of fibres within such composites



2 Single fibre tensile testing set-up

tends to be similar to that in air. The focus of the current work is therefore not on oxidation of a large, flat plate of the stainless steel concerned, but rather on the durability in air of these particular fibres (with a specific diameter, surface roughness, etc., as well as a specific composition). There is interest, for example, in the proportion of metal oxidised during a particular heat treatment. Oxidation kinetics data are therefore presented, not only in terms of the relative weight change, but also as a residual fraction of metal. Since the volume (and mass) of both metal and oxide is changing during oxidation, this requires assumptions concerning the oxidation reaction (which in practice could involve the formation of Ni, Fe or Si oxides, as well as Cr_2O_3). In the present work, it has been assumed that the reaction can be represented as follows

$$2\text{M (molecular mass} = 112) + \frac{3}{2}O_2 \rightarrow \quad (1)$$
$$M_2O_3 \text{ (molecular mass} = 160)$$

so that 1 g of metal yields 1·429 g of oxide. This relationship allows measured weight gains to be converted to weight of metal lost, and hence to residual weight fraction of metal. It is not, of course, highly accurate, but is nevertheless considered useful for present purposes.

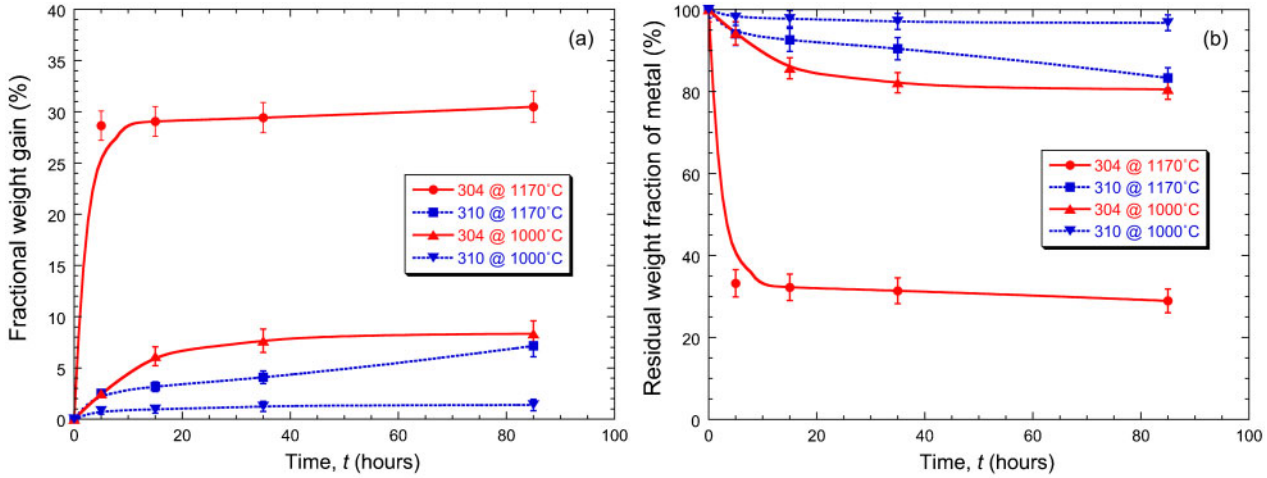
Single fibre tensile testing

Fibres of length 25 mm were heat treated in a similar manner to the oxidation testing described above, except that they were in open crucibles, rather than ampoules. After the heat treatment concerned, fibres were mounted in a screw driven loading system, with a 500 N load cell. The set-up is illustrated in Fig. 2. Displacements were measured using a scanning laser extensometer, having a resolution of ~3 μm. Gauge lengths (of ~8 mm) were defined by two 'flags' glued to the sample, which interrupted the scanning laser beam. Wire grips were employed, and the displacement rate was 1·0 mm min^{-1} in all cases.

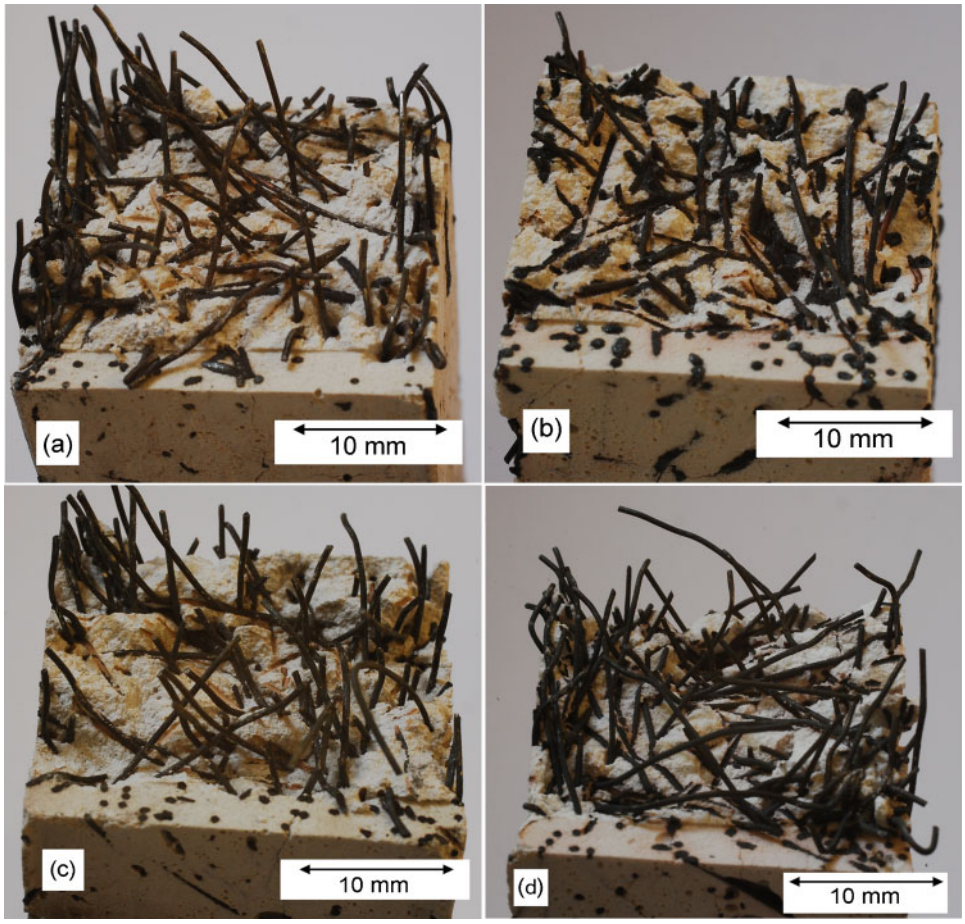
Composite fracture energy measurement

Impact tests were conducted using a conventional pendulum based 160 J Izod testing rig, with specimen dimensions being $30 \times 30 \times 100$ mm in all cases. They were prenotched, to a depth of 4 mm, at a suitable location, using a diamond saw with a width of 0·6 mm. After fracture, the energy absorbed is obtained from a pointer on the machine, based on the height of the pendulum before and after impact. The fracture energy G_c was then obtained on dividing this energy by the residual cross-sectional area at the notch. There is an implicit assumption that the fracture occurred under mode I (crack opening) conditions, in plane strain. In practice, neither condition is likely to have been rigorously met, particularly towards the end of fracture.

Cases where the fracture did not initiate at the notch were removed from the data set. (This occurred only in a small number of cases.) In general, fracture was confined to a reasonably well defined crack plane, although there was certainly some associated fragmentation of the matrix. Typical fracture surfaces are shown in Fig. 3, where it can be seen that pullout and/or rupture of fibres bridging the crack plane were common and extensive. However, it can also be seen that fibre oxidation has a pronounced effect on the fracture behaviour. For



3 Weight gain data from heat treatment in air of 304 and 310 samples at 1000 and 1170°C, plotted as *a* fractional weight gain and *b* residual weight fraction of metal, obtained using equation (1)



a 304, 1000°C; *b* 304, 1170°C; *c* 310, 1000°C; *d* 310, 1170°C

4 Photomicrographs of fracture surfaces after Izod testing of composite materials containing following types of fibre, after 20 h heat treatment at indicated temperature

example, the fibres were clearly rather ineffective in bridging the crack plane in the case of the 304 reinforced composite heat treated at 1170°C.

Fibre oxidation characteristics

Data from the oxidation study are presented in Fig. 4. These are presented both as fractional weight gains and as residual fraction of metal. The latter plot is helpful, since it indicates whether an observed fall off in oxidation rate might be largely associated with the decreasing residual area of metal available for oxidation. Clearly, this is more likely for smaller diameter fibres. It can be seen in this figure that, as expected, the 304 fibres oxidise much more rapidly than the 310 fibres. In fact, it is clear that, at a temperature of 1170°C, a short period of a few hours is sufficient to oxidise most of the section of a 304 fibre. Even at 1000°C, these 304 fibres oxidise at a rate sufficient to reduce the residual metal section considerably within a few tens of hours. In contrast, the 310 fibres are considerably more resistant to oxidation and much of the metal section apparently remains intact even after very substantial heat treatments, such as a period of the order of 100 h at 1170°C.

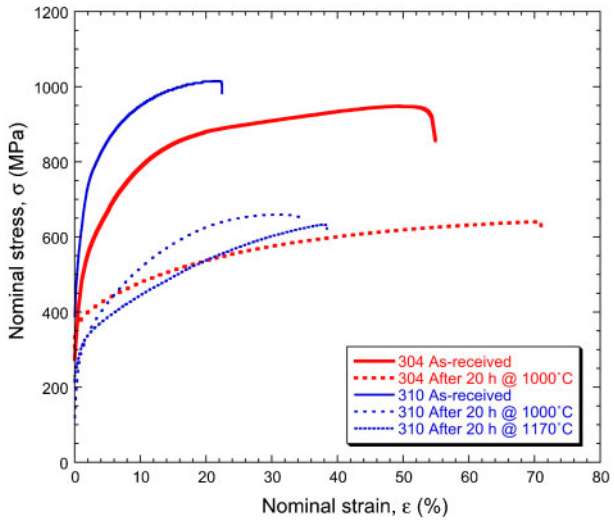
Comparison with previously published oxidation rate data can only be very limited, since the current results are specific to these fibres. Nevertheless, it is clear that the superior performance of the 310 is consistent with extensive information in the literature. It is only possible to make approximate comparisons with actual oxidation rates (in mg cm^{-2}), mainly because of the geometrical effects associated with using (relatively small diameter) fibres, but in general the results reported here appear to be broadly consistent with previously reported values for flat plates.[7]

Effect of heat treatment on mechanical properties

Fibre tensile characteristics

Representative stress–strain plots from the tensile tests are shown in Fig. 5. Several features are apparent. The as received (annealed) 304 fibres exhibited a ductility of about 50–60% and a strength (flow stress) of ~800 MPa. These values are in the range broadly expected of 304 in this type of condition. This is also true for the as received 310 fibres, which are, if anything, slightly stronger, but have a lower ductility (of around 20–30%). Heat treatment at 1000°C for a relatively short period of 20 h reduced the strength in both cases to ~600 MPa while increasing the ductility (to about 60–70% for 304 and 35–40% for 310). This is also broadly as expected, with the heat treatment presumably causing a certain amount of grain growth and reduction in dislocation density. It is clear from Fig. 3 that this heat treatment does not cause excessive oxidation for either type of fibre.

However, the behaviour of the two types of fibre diverges sharply after more severe heat treatments. For



5 Representative stress–strain plots for 304 and 310 fibres, before and after heat treatment

the 310 fibres, a similar ductility and strength are retained even after 20 h at 1170°C. In contrast, it was found that the 304 fibres became so brittle after this heat treatment (and also after 20 h at 1100°C) that they could not be tested. This is consistent (for the 1170°C case) with the oxidation behaviour shown in Fig. 3, in the sense that this heat treatment converted so much of the fibre to oxide that there would be little or no residual metal to bear any mechanical load.

Data from individual fibre tensile tests are shown in Fig. 6. These data are in the form of the work of fibre deformation (area under the stress–strain curve) and the strain to failure. It can be seen that these plots are consistent with those in Fig. 5. These two parameters have been highlighted in view of their role in the previously developed model[1] for prediction of the fracture energy of MFCs (*see* the section 'Composite fracture energy').

Composite fracture energy

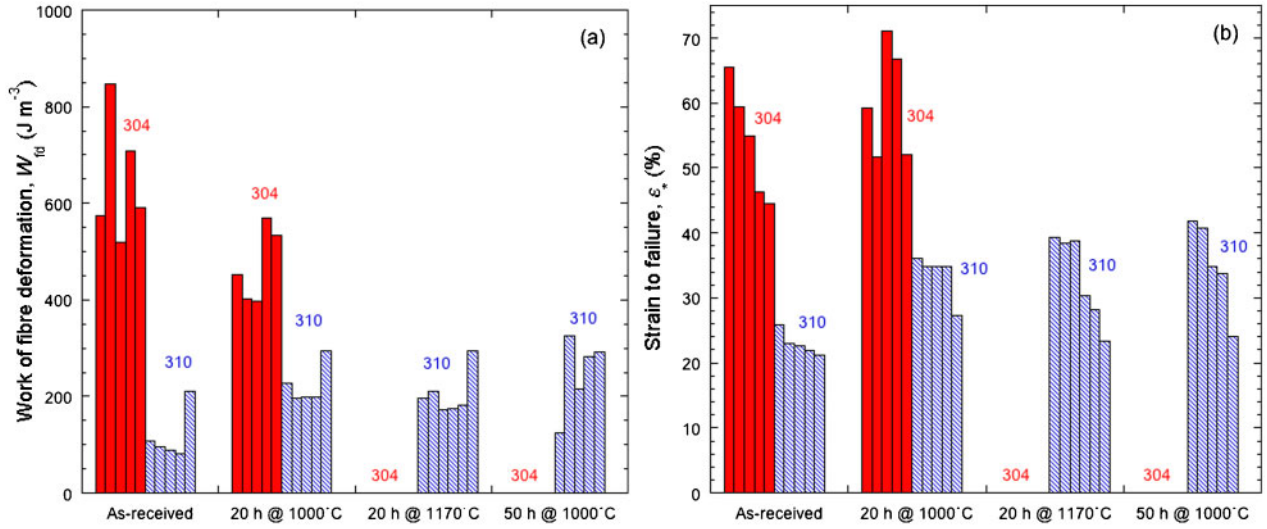
Data from the Izod testing are presented in Fig. 7. It may first be noted that these fracture energy values (of the order of several tens of kJ m^{-2}) represent excellent toughness levels (comparable with those of many metals). It can also be seen that, for the composite samples in the as produced state, i.e. the fibres in the as received condition), the toughness of 304 containing material is a little higher than that of samples reinforced with 310 fibres. However, it is clear that the effect of heat treatment is very different in the two cases, with the initial toughness level being largely retained with 310 reinforcement, but falling rather sharply with increased severity of the heat treatment for composites containing 304 fibres.

The behaviour represented in Fig. 7 is qualitatively consistent with the observed effect of these heat treatments on single fibre characteristics, in the sense that the degradation of composite toughness is much sharper in the case of 304 reinforcement, as is the impairment of tensile properties (and the rate of consumption of the metal by oxidation). Moreover, it is possible to quantify the relationship between the two using the model[1] for prediction of the fracture energy of the composite. In summary, the model is based on quantification of the energy absorbed by pullout and plastic deformation and rupture of fibres bridging the crack plane. The net work of fracture is estimated by summing the two contributions, assuming that a fraction g of bridging fibres undergo pullout and the remainder $(1-g)$ undergo plastic deformation and rupture, leading to the following equation

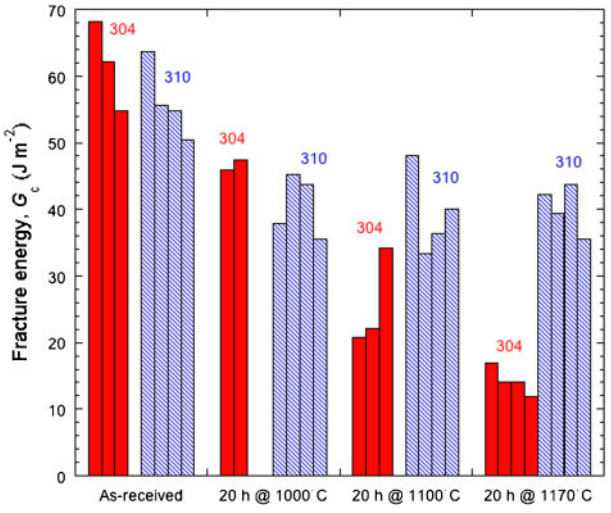
$$G_{\mathrm{cnet}} = gG_{\mathrm{cpo}} + (1-g)G_{\mathrm{cfd}} = g\frac{fs_{\mathrm{po}}^2 R\tau_{\mathrm{i}*}}{2} + (1-g)\frac{fs_{\mathrm{fd}} RW_{\mathrm{fd}}}{\varepsilon_*} \quad (2)$$

where f is the fibre volume fraction, s_{po} is the aspect ratio of pulled out fibres protruding from the crack plane, s_{fd} is the corresponding aspect ratio for plastically deformed fibres, R is the fibre radius, $\tau_{\mathrm{i}*}$ is the shear stress for frictional sliding during pullout, W_{fd} is the work of fibre deformation (per unit volume) and ε_* is the strain to failure.

The parameters required for implementation of equation (2) include the protruding fibre aspect ratios,



6 *a* work of fibre deformation (area under tensile testing stress–strain curves) and *b* tensile strain to failure for individual 304 and 310 fibres before and after heat treatment



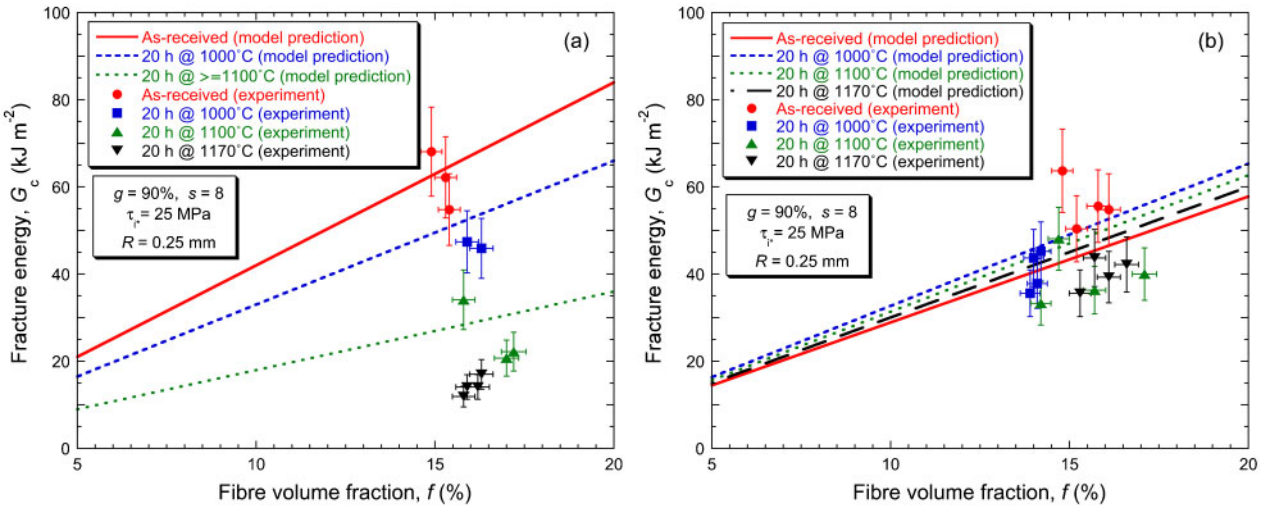
7 Fracture energy values obtained from Izod testing of as received and heat treated composite samples containing (~15 vol%) 304 or 310 fibres

although it is not easy to distinguish fibres that have undergone pullout from those that have experienced extensive plastic deformation. By inspection of fracture surfaces such as those shown in Fig. 3, it was decided that ascribing a value of 8 to both would be a reasonable approximation. (Since the initial fibre aspect ratio is ~50, i.e. 25 : 0·5, the value of s can be capped at about half of this, although in practice the approximately random fibre orientation distribution ensures that most protruding fibres will have an aspect ratio well below this limit.) The shear stress for frictional sliding has not been measured for the fibres being used in the current work, but it was estimated previously[1] to be ~25 MPa for melt extracted fibres, and this value has been employed here. The appropriate values of W_{fd} and ε_* are those from tensile testing of the fibres concerned: average values were taken in particular cases from sets of experimental data such as those presented in Fig. 6.

The outcome of the operation of obtaining predicted work of fracture values for composites subjected to particular heat treatments, and comparison with corresponding experimental data, is shown in Fig. 8. (The fibre volume fraction values used to plot the individual experimental points were obtained from the samples concerned by density measurement.) It can be seen that, for both Fig. 8, i.e. for both 304 and 310 fibres, there is reasonably good agreement between predicted and measured fracture energy values, for a range of heat treatments. For 304 fibres, progressive oxidation and embrittlement as the heat treatment severity is increased, reflected in the tensile test data, lead to a decrease in the predicted composite toughness, which is consistent with the experimental Izod measurements. On the other hand, retention of good tensile test properties by 310 fibres after the same heat treatments is reflected in a prediction that high composite toughness will also be retained, which is again consistent with experiment.

It is worth noting that there are no arbitrarily adjustable parameters in the formulation of the model. The variable that presents the most difficulty in evaluating is g, i.e. the proportion of fibres (bridging the crack plane) that undergo pullout, rather than plastic deformation. As emphasised in the previous publication,[1] the latter mechanism commonly has the greater capacity for energy absorption (particularly for fibres with a large value of W_{fd}), so any measure that tends to promote it, and hence reduces the value of g, is likely to be beneficial. However, it was concluded in the initial work[1] that, typically, the effective value of g is ~90%, and this is the value used in Fig. 8.

Overall, there is a clear message from the information presented here that, for components requiring good mechanical properties during and after severe heat treatments, the use of 310 fibres would be recommended in preference to 304 (and also in preference to most other stainless steels). There are several other microstructural features that can be optimised in terms of composite toughness with the assistance of the model. These include fibre diameter (and aspect ratio), fibre volume fraction, the presence of hooks at fibre ends and the interfacial bond strength. Some of these issues (notably the first two) were at least provisionally addressed in the previous publication,[1] and others are deferred to future work.



8 Predicted and measured (Izod) fracture energies, as function of fibre volume fraction, before and after heat treatment, for composites reinforced with *a* 304 fibres and *b* 310 fibres

Conclusions

The following conclusions can be drawn from this work.

1. It has been confirmed that 310 fibres (with a diameter of 0·5 mm) exhibit considerably better resistance to oxidation in air than 304 fibres of the same size. In particular, while 310 fibres remain largely intact after 100 h at temperatures as high as 1170°C, 304 fibres are entirely consumed by such a treatment and indeed become substantially oxidised after a few tens of hours at 1000°C.

2. Tensile tests carried out on these fibres before and after various heat treatments have confirmed that, while moderate treatments can lead to increased ductility, exposure to high temperature (sufficient to cause significant oxidation) leads to impaired mechanical properties, in the form of reductions in both strength and strain to failure. There is thus far superior retention of original properties for 310 fibres than for 304 fibres. The latter are too brittle and weak to be tested after a few tens of hours at 1000°C, or shorter periods at 1100°C. On the other hand, the 310 fibres retained good strength and ductility after a period of the order of 100 h at 1170°C.

3. The focus of the current work is on the toughness of ceramic matrix composites reinforced with these fibres (~15 vol%, with an approximately isotropic orientation distribution), before and after heat treatment. This has been measured using Izod impact testing. The values obtained are unlikely to be highly accurate, but are considered useful for exploring various trends and sensitivities. It has been found that, while as produced composites containing 304 fibres exhibited slightly higher fracture energies than those containing 310 fibres (both ~50–60 kJ m^{-2}), this fell rapidly with 304 reinforcement on increasing the severity of the heat treatment, but was largely retained for the 310 composites. This is clearly consistent with the concept of the mechanical properties of the fibre playing a key role in dictating the toughness of the composite.

4. Quantitative correlation between mechanical properties of the fibre and composite toughness has been explored using a previously presented model, based on the work of fracture being dominated by contributions from fibres bridging the crack plane (and experiencing frictional pullout and/or plastic deformation). Good agreement is observed between predicted and observed values, particularly in terms of general trends. The main value of the model probably lies in highlighting various sensitivities. A key point is that there is greater potential for energy absorption from fibre plasticity than from fibre pullout, provided the work of fibre deformation (expressed per unit volume) is relatively large, i.e. provided the fibre exhibits good strength and ductility. This model thus provides a quantitative framework for exploring various effects, including the influence of fibre type and heat treatment described here.

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