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Effect of sodium on the creep resistance of yttrium aluminium garnet (YAG) fibres

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Abstract

Aligned YAG fine fibres had been made previously from an aqueous sol–gel process, but it was suspected that sodium contamination in one of the starting materials lessened the creep resistance of the final product. Therefore, sodium-free gel precursor fibres were made using the same process, and upon firing in air at 900 °C these formed pure phase YAG fibres, of 4 μm diameter. When steamed over 200–500 °C for 3 h, the amorphous gel fibre formed single phase nanocrystalline YAG between 400 and 500 °C, an extraordinarily low temperature for this material to crystallise. Upon postfiring up to 1550 °C, grains averaging 0.5 μm and pores of 0.17 μm had formed, but despite this porosity and smaller grain size, the sodium-free fibre exhibited superior creep resistance than the sodium contaminated fibres reported previously by the authors, typically creeping at temperatures 50 °C higher. This demonstrates that even small levels of sodium contamination are harmful to creep resistance in YAG.

Keywords: Yttrium aluminium garnet; Fibres; Creep; Sol-gel processes; Microstructure-final

1. Introduction

To overcome the problem of inherent brittleness in ceramic materials, continuous fibres can be used as a reinforcement in the ceramic matrix, particularly in high temperature applications over 1400 °C. Yttrium aluminium garnet (YAG, $Y_3Al_5O_{12}$) is a suitable candidate fibre, due to its high thermo-mechanical stability, resistance to both oxidising and reducing atmospheres at high temperatures, and a melting point of 1970 °C.[1] Of all the ceramic materials currently investigated, single crystal YAG also demonstrates the best all-round resistance to creep,[2] exhibiting a creep resistance 10 times higher than single crystal alumina.[3] In polycrystalline YAG lattice and boundary diffusion will reduce the creep resistance of the material, but nevertheless, at 1400 °C polycrystalline YAG with a grain size of 3 μm is three times better than polycrystalline alumina of the same grain size.[4] Furthermore, the creep in YAG is very weakly dependent upon orientation,[5] unlike the strongly anisotropic creep in alumina, removing the complication of crystal alignment from any future manufacturing process. Although the exact mechanism of creep in single crystal YAG is still uncertain, it is suggested that it is largely a diffusion-controlled process,[6] as the large size of the cubic unit cell and large lattice parameters of the garnet dramatically reduces the number of dislocations, especially climb dislocations which are a key creep mechanism.[7]

In previous publications we have reported the synthesis and characterisation of a stable, aqueous sol–gel YAG precursor,[8] produced from a stoichiometric combination of a commercially available aluminium sol (Hoechst *Locron-L*) and an aqueous yttria sol. This was then spun to give a gel-fibre product, which upon heating resulted in an aligned blanket of YAG continuous fine fibres, with diameters of only 5.5 μm. These fibres were produced by a modified commercial blow spinning process,[9,10] a notable feature of which was

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that the gel fibres were generated at 9 ms^{-1} and set within 1 s. This gel fibre underwent a subsequent crystallisation from amorphous material to single phase YAG fibres between 700 and 800 °C, the lowest reported crystallisation temperature for this material, and this indicated that the sol was an extremely homogenous mixture. This YAG fibre had a grain size of less than 0.1 μm which increased to only 1 μm when fired to 1550 °C for 3 h, but it remained 20% porous even at this temperature, resulting in a material with poor mechanical properties. These aligned textile-dimension fibres were a superior fibrous material to those reported by other workers, and they were manufactured at a faster spinning rate in an already-dried state on equipment suitable for future industrial development. Unfortunately, to optimise the creep resistant properties of YAG it is important that the fibres are well sintered and contain no defects likely to cause loss of mechanical strength.

It was demonstrated that if a yttria sol containing chloride counterions was used instead, the precursor was stable at higher concentrations, and this resulted in a denser gel fibre.[11] The chloride containing fibres showed improved sintering characteristics, being 95% sintered at 1400 °C and 98.5% at 1550 °C, whereas the nitrate containing fibres had given no indication of nearing maximum density. However, when fired in air the amorphous fibres did not become fully crystalline until 900 °C, a temperature 100 °C higher than in the fibres made from the yttria-nitrate sol. There was evidence that chlorine retained in the fibre up to this temperature delayed the formation of the YAG phase, a feature also observed in chloride bearing hexagonal ferrite fibres.[12] The chlorine was also responsible for causing many hemispherical faults to form in the fibre, thus drastically reducing its mechanical strength, and these faults had to be removed to render the fibres useful. This was achieved through the steaming of the fibres at lower temperatures, before the formation of the YAG phase. Fibre steamed from 200 to 500 °C over 3 h formed an amorphous product, which produced crystalline YAG upon subsequent firing in air to 800 °C, with no hemispherical surface faults present, improving the mechanical properties of the fibre.[13] Once YAG had formed the sintering and grain growth of the steamed fibre proceeded just as before.

The creep behaviour of the 1550 °C fired fibres was similar to that of coarser polycrystalline YAG fibres when allowance was made for grain size, but surprisingly a ten-fold reduction in grain size when the fibre was sintered at 1400 °C increased the creep rate by only 50%.[11] This was speculatively attributed to the presence of sodium as a mobile grain boundary impurity, as any sodium will be more dispersed in the finer grained fibre and its affect on creep rates will be reduced. The *Locron-L* precursor was made from sodium aluminate, containing 130 ppm of sodium to one part of alumina,[14] and this was the only source of sodium contamination. On the surface of a 1 μm grain, the sodium level of approximately 300 ppm on YAG corresponded to about 4.8 Na atoms/nm^2 or complete interfacial coverage by $10Al_2O_3{\cdot}Na_2O$. Therefore, this paper investigates the production of a sodium-free YAG fibre, using a sodium-free source of alumina, and compares the resulting fibres to those previously manufactured.

2. Experimental

2.1. Preparative methods

2.1.1. Sol preparation

Previously the source of alumina was Hoechst *Locron-L* aluminium chlorohydrate solution, containing 23.5% alumina by weight, which is manufactured from sodium aluminate. For this work a commercial source of sodium-free aluminium chlorohydrate was identified, made from aluminium sulphate. This was Reheis *Chlorohydrol* solution, containing 23.2% alumina by weight, with a ratio of Al:Cl of 1.97, and the only significant contaminant was stated to be 36 ppm of iron.[15] The yttria sol was made as before,[11] from the peptisation in 0.5 M hydrochloric acid (yttrium:acid ratio = 2:1) of yttrium hydroxide precipitated from yttrium chloride. Typical assays of the yttrium chloride (99.99% purity) were obtained from the manufacturer,[16] and consisted of rare earth metal impurities totalling 600 ppm, with Si, Mg, Al, Ca, Pb, Fe and Zr at levels of 70, 20, 20, 20, 20, 2 and 1 ppm by weight, respectively. The two components were then combined in stoichiometric amounts to form a mixed precursor sol.

2.1.2. Blow spinning

The mixed sol was filtered through a 0.7 μm filter, and then rendered spinnable by the addition of a small amount of polyethylene oxide (PEO) as a spinning aid, followed by further concentration. The fibres were produced by a modified proprietary blow spinning process[9,10] in which the spinning solution was extruded through a row of holes, on either side of which impinge parallel jets of humidified attenuating air. The fibres were gelled by mixing in a stream of hot secondary air at 100 °C and then collected as a continuous, dried gel fibre in aligned form on a drum rotating at 1000 rpm. A notable feature of the gel and spinning process was that the fibres were generated at 9 m/s and set within 1 s. After collection the fibres were removed and stored in a circulating oven at 110 °C to await subsequent heat treatment.

2.1.3. Heat treatments

The fibres were steamed to remove the chloride before firing to higher temperatures, using the optimum regime used previously for the YAG fibres.[13] They were heated to 200 °C at 100 K/h in air, then steamed from 200 to 500 °C over 3 h at 100 K/h. The fibres were steamed in a fused silica tube furnace with a volume of 1018 cm^3. Water was injected into the tube through a *Saffil* wick at a rate of 18 ml/h, which provided a gas flow of 38,744 cm^3/h at 200 °C, so the tube was flushed at least every 95 s. After steaming the fibres were allowed to cool down in air, and then further heated at 300 K/h to the desired temperature, where they were maintained for 3 h.

2.2. Characterisation

2.2.1. X-ray powder diffraction (XRD) measurement

X-ray powder diffraction patterns of the samples treated at various temperatures were recorded in the region of $2\theta = 10–80^\circ$ with a scanning speed of 0.25°/min on a Philips PW1710 diffractometer using Cu Kα radiation with a nickel filter. Philips APD 1700 software was used to calculate the average size of the crystallites in a sample using the Scherrer equation:

$$D = \frac{K\lambda}{h_{1/2}\cos\theta}$$

where D is the average size of the crystallites; K, Scherrer constant (0.9×57.3); λ, wavelength of radiation (1.5405 Å); $h_{1/2}$, peak width at half height and θ corresponds to the peak position.

2.2.2. Scanning electron microscopy (SEM)

Scanning electron micrographs and analysis of the morphology of the samples was carried out on a Cambridge Instruments Stereoscan 90 SEM operating at 10 kV. Conducting samples were prepared by gold sputtering fibre specimens. Approximate grain sizes were obtained from SEM micrographs, simply by measuring all the grains in a horizontal line, and taking an average.

2.2.3. Creep measurements using the bend stress relaxation (BSR) technique

The bend stress relaxation (BSR) technique was developed by Morscher and Dicarlo[17] and has been used in other work[5,11] to rank the creep performance of polycrystalline YAG fibres. The test was carried out on strips of aligned YAG fibre 1 mm × 10 mm, with the longer dimension parallel to the axis of alignment. These fibres were prefired to a nearly sintered state, so any observed effect was due to creep, and not a sintering effect. For this the fibres were sintered at 1400 and 1550 °C/3 h, before creep testing at temperatures for 1 h, and no change in grain size or was observed after testing. The fibres also showed no further shrinkage at 1350 °C/h, after prefiring at 1400 or 1550 °C/3 h.

In the test the fibre was sandwiched between a 4 mm alumina rod (0.1% strain for a 4 μm fibre) resting on two other larger rods of 8 mm diameter (Fig. 1). The fibre, was therefore, bent to a known initial diameter, d_0 (= 4 mm), imposing a surface strain given by d/d_0 where d is the fibre diameter. The fibre was then heated to various temperatures where it was held for 1 h, and then allowed to cool down immediately in air to room temperature and the strain-inducing rod removed. If a stress relaxation has taken place and creep has occurred the fibre retains a residual curvature of diameter df.

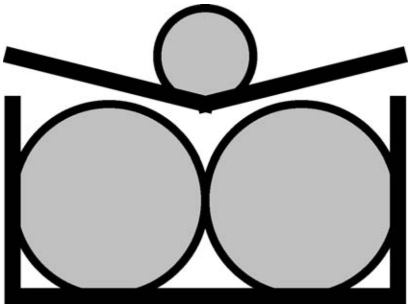

Fig. 1. Cross sectional diagram of the fibre-alumina rod sandwich in BSR test.

The stress relaxation ratio, M, can then be calculated from the equation:

$$M = 1 - \frac{d_0}{d_\mathrm{f}}$$

If $M = 1$ this means that no creep has taken place, while $M = 0$ indicates that the fibre has undergone total stress relaxation and crept around the diameter of the rod. These results can then be plotted against temperature and compared to previous results. The rod size was chosen so that even the smallest fibres (3 μm diameter) were subjected to no more than 0.15% strain, and the diameter of creep could be measured with a reasonable degree of accuracy (±0.4 mm). Morscher indicated that during the BSR test a sample undergoes a creep strain proportional to $(1/M) - 1$, so that the technique may be used to make approximate quantitative comparisons. Although this test is only an approximate measurement of creep, the YAG fibres compared here were all measured by the same BSR technique.[5,11]

3. Results and discussion

3.1. Spinning of gel fibres

The change of aluminium chlorohydrate appeared to have little effect on the particle size distributions of the precursor sol, which resembled those of the sodium contaminated YAG precursors.[8,11] However, upon concentration of the spinning solution after addition of PEO, the maximum concentration achievable was somewhere between the values of the chloride stabilised sol (>0.84 mol/l YAG) and the nitrate stabilised sol (0.56 mol/l YAG) reported previously. The sol had gelled to a dry solid at 0.7 mol/l YAG, although it was easily redispersed and spun following the addition of a few drops of water. Therefore, although the sol was stable, the *Chlorohydrol* solution appeared to be interacting with the sol more compared to *Locron-L*, resulting in gelling at lower concentrations. This sol was also more difficult to spin, exhibiting higher viscoelasticity, again an indication that the *Chlorohydrol* solution was causing more structuring within the sol than seen with *Locron-L*. However, smooth, parallel sided gel fibres were still produced, with diameters between 3 and 5 μm, and as before the fibres were well aligned.

3.2. Heat treatment of fibres

As observed previously in the YAG fibres, they had to be fired to over 800 °C in air to form the pure YAG phase, which had a crystallite size of 26 nm at 900 °C, and no discernible grain structure using SEM. However, the hemispherical pits seen before were also present, and thus the fibres needed to be steamed prior to YAG formation to remove the chloride. On steaming under the optimum regime (200–500 °C over 3 h) discovered previously[13] it was found the fibres had produced pure phase YAG at only 500 °C. This is an extremely low temperature for YAG to form at, being even 300 °C lower than the lowest crystallisation temperature previously reported by the authors.[8] As can be seen in Fig. 2b, the fibres were well crystalline at this temperature, with a calculated crystallite size of 20 nm, similar to the YAG fibres formed previously at 800–900 °C when fired in air. As can be seen in Fig. 3, the steamed YAG fibres were featureless with no grain structure apparent. When the fibres were steamed to only 400 °C they resembled the previous samples when steamed to 500 °C, showing only an amorphous hump between 20 and 40° (Fig. 2a), and the same amorphous XRD pattern was seen when the sample was fired to 500 °C in air.

The steamed fibres were then heated in air to 1400 and 1550 °C for creep testing, and the average fired YAG fibre diameter was 4 μm. As the XRD patterns in Fig. 2c and d show, there was no change in phase, and the YAG became more crystalline with increasing temperature. The crystallite sizes, calculated to be 62 nm after firing to 1400 °C and 84 nm after 1550 °C, were larger than in the sodium contaminated YAG fibres, which were only 68 nm at 1550 °C. The morphology of the sodium-free fibres was very different however. At 1400 °C the previous YAG fibres still had no apparent grain structure, whereas the sodium-free fibres had grains with a diameter of around 250 nm (Fig. 4). They were also full of regularly shaped pores between 80 and 130 nm in diameter, which were never observed in any of our previous YAG fibres, their porosity being estimated from shrinkage data. The surface area of the YAG fibres was too small to be accurately measured by nitrogen adsorption techniques, and the linear shrinkage of the sodium-free fibres was similar to that of the other steamed YAG fibres containing chlorides. With further

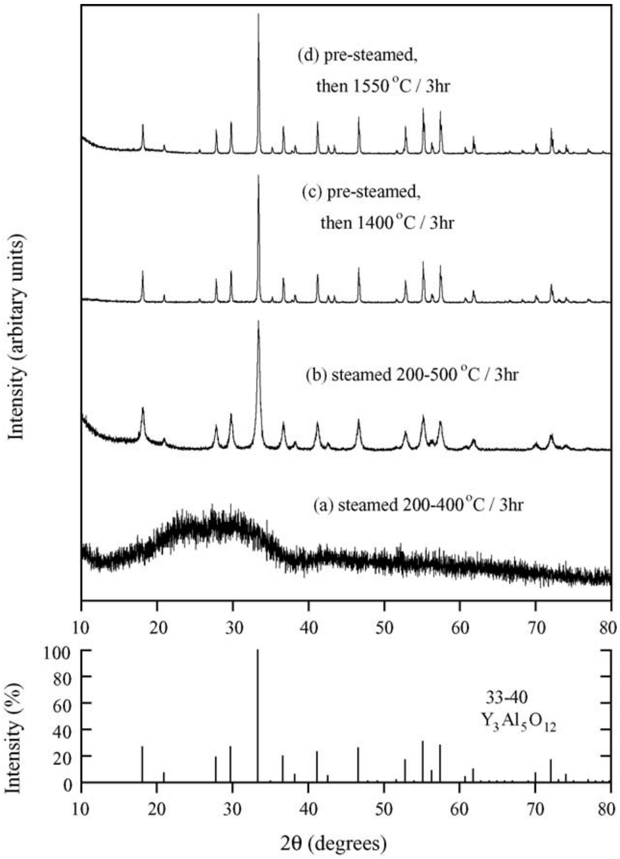

Fig. 2. XRD patterns of YAG fibres steamed at: (a) 200–400 °C and (b) 200–500 °C over 3 h, and following (b) then heated in air at (c) 1400 °C and (d) 1550 °C for 3 h.

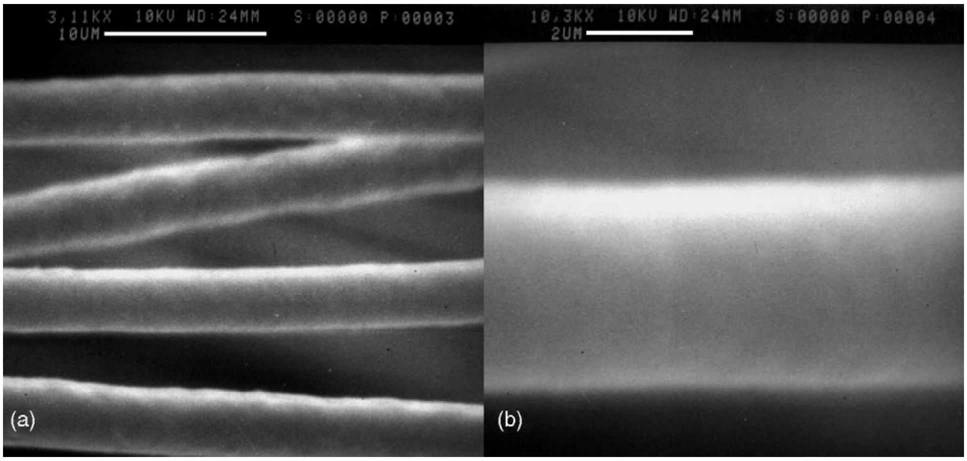

Fig. 3. SEM micrographs of YAG fibres steamed at 200–500 °C over 3 h, taken at: (a) 3k and (b) 10k magnification.

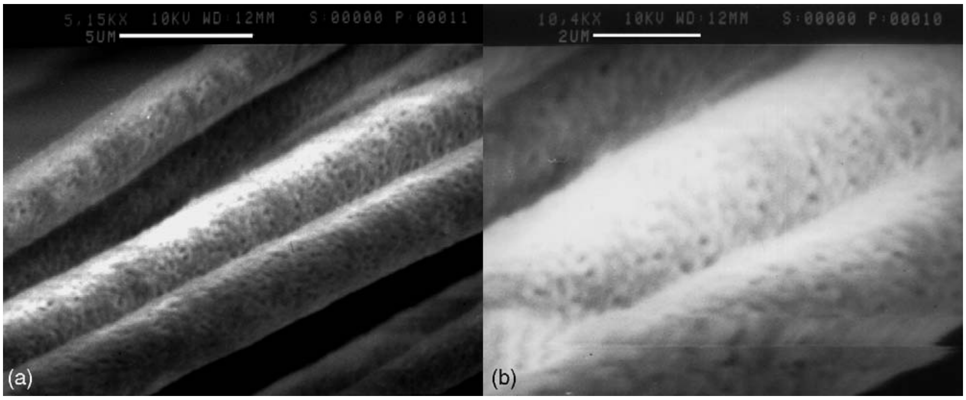

Fig. 4. SEM micrographs of YAG fibres fired to 1400 °C/3 h, taken at: (a) 5k and (b) 10k magnification.

heating to 1550 °C many grains had grown, as had the diameters of the pores, but there were now less pores visible on the surface of the fibres (Fig. 5). Both pores and grains appeared to be regularly shaped, with a pore diameter of 150–170 nm, and the almost bubble-like grains measuring between 250 and 600 nm, with an average diameter of around 500 nm. While all the grains were smaller than the 1 μm grains observed in the sodium contaminated fibres at this temperature, the obvious porosity of the fibres would be expected to mechanically weaken the sodium-free YAG fibres.

3.3. Creep resistance of sodium-free YAG fibres

Creep was assessed for the sodium-free YAG fibres prefired to 1400 and 1550 °C using the BSR technique, firing for 1 h at 50 K increments between 1000 and 1400 °C. The results are compared to those for the sodium contaminated YAG fibres in Fig. 6. It can be seen that both the sodium-free fibres creep at temperatures approximately 50 °C higher than the previous YAG fibres, so that the sodium-free fibres fired to 1400 °C almost shadow the contaminated fibres fired to 1550 °C. Although all the curves generally show similar shapes, both sodium-free fibres resist creep better

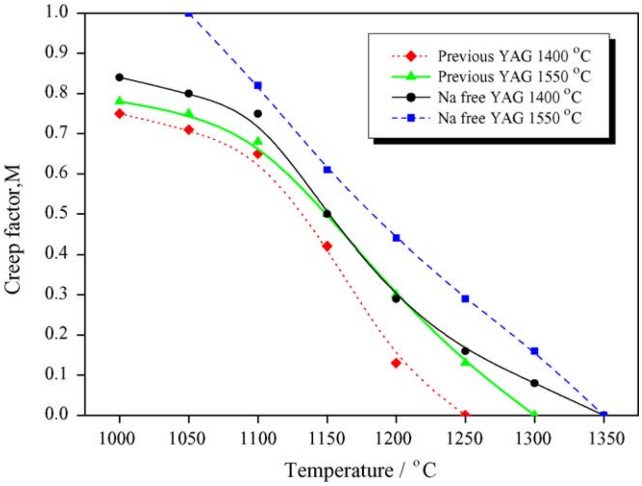

Fig. 6. Comparison of the creep of sodium-free YAG fibres, prefired to 1400 and 1550 °C/3 h, over the temperature range 1000–1350 °C, and previously reported polycrystalline YAG fibres manufactured by the authors from the same process.[11]

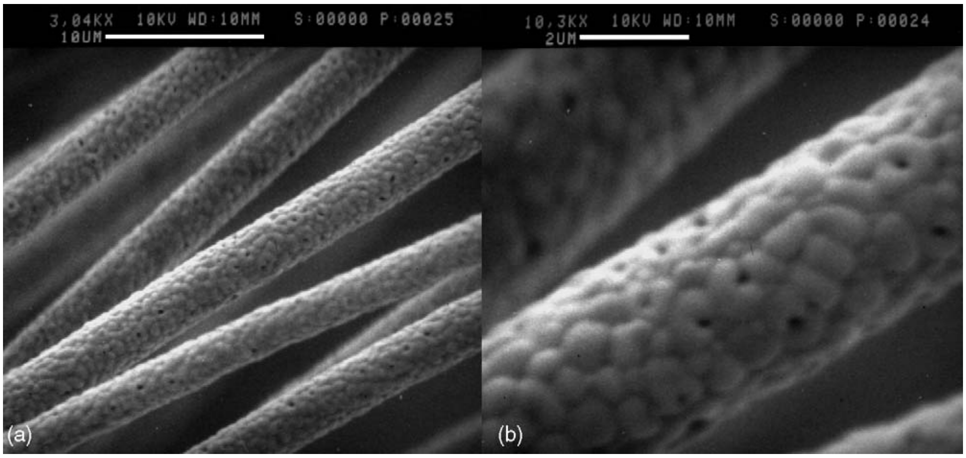

Fig. 5. SEM micrographs of YAG fibres fired to 1550 °C/3 h, taken at: (a) 3k and (b) 10k magnification.

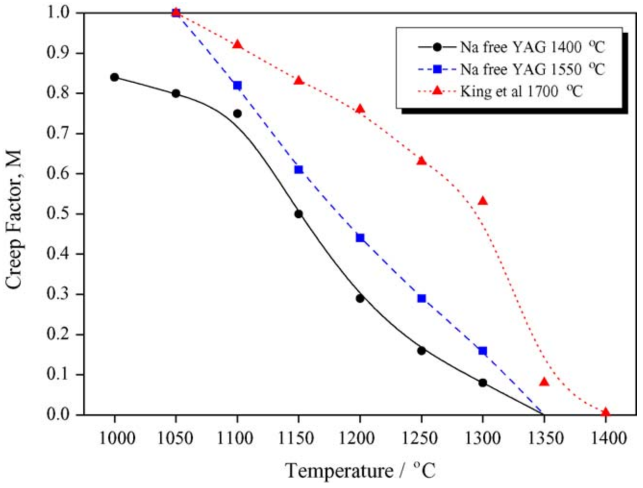

Fig. 7. Comparison of the creep of sodium-free YAG fibres, prefired to 1400 and 1550 °C/3 h, over the temperature range 1000–1350 °C, and previously reported polycrystalline YAG fibres prefired to 1700 °C.[5]

at higher temperatures, and the 1550 °C fibre is markedly superior at lower temperatures, showing no sign of creep at all below 1100 °C. This was despite the porosity observed in the sodium-free YAG fibres and the smaller grain size of the 1550 °C fired fibre, both of which would lower creep resistance, demonstrating that the presence of sodium on the surface of grains is indeed harmful to creep resistance.

In Fig. 7 the sodium-free fibres are compared to the polycrystalline YAG fibres reported by King and Halloran,[5] which were prefired to 1700 °C and also were tested using the BSR technique. Although King's fibres still outperform the sodium-free YAG fibres between 1100 and 1300 °C, the sodium-free YAG fibres prefired to 1550 °C do not begin to creep until the same temperature, and both fibres come close to King's YAG fibre at 1350 °C. However, it must be noted that the measurements in[5] are likely to be more accurate as they are made on 120 μm rather than 4 μm diameter fibres. Also in the present work we were obliged to use radius templates to make undistorted measurements of curvature on tape samples from fibre blankets, so that the measurements carry an uncertainty of ±0.4 mm, due to the steps between template sizes. This uncertainty amounts to ±10% as the fibre becomes fully strained. Quantitative comparisons of creep rate also become unrealistic as M approaches 0, as the error in the relative creep strain ($1/M - 1$) is greatly magnified, but a reasonable comparison can be made down to $M = 0.3$ in this work. This would indicate that at temperatures of 1100, 1150, 1200 and 1250 °C, the 1550 °C sodium-free YAG fibre crept 2.4, 3.8, 4.9 and 3.5 times faster, respectively, than King's fibre—an improvement upon the sodium contaminated fibre investigated previously.

If the creep process in YAG is considered to be mostly a diffusion driven process, we can assume the grain size to be the deciding factor in the difference between the creep resistance of the polycrystalline fibres. The King fibre had a grain size of 3 μm, compared to 0.5 μm for the 1550 °C sodium-free YAG and 0.25 μm for the 1400 °C sodium-free YAG fibre. Whilst to maintain the strength and flexibility of the fibre it is preferable to keep the grain size as small as possible, and certainly below the submicron level, for the purposes of good creep properties it is preferable to have the grains as large as possible, to limit the possibility of grain-boundary diffusion. Indeed, with his large diameter fibres King has recommended either an increase in grain size or an axial elongation of the grains to suppress diffusional creep.[18] However, to balance the requirements of creep resistance against mechanical strength a grain size of 0.5–2 μm is claimed to be ideal,[19] and the sodium-free YAG fibres clearly satisfy this requirement. The effects of grain size on creep rate are well reported in the literature with rates proportional to $1/d^2$ in a system controlled by Nabarro–Herring lattice diffusion, and proportional to $1/d$ where grain boundary sliding is a predominant mechanism (assuming there is no second phase at the boundary). Fig. 8 illustrates that the difference in M between King's fibres with 3 μm grains and both our 1400 and 1550 °C prefired sodium-free YAG fibre can be attributed to differences in grain size, whichever detailed mechanism applies, and in reality creep is probably controlled by a combination of the two. Firing temperatures need to be extended to 1700 °C in future work to match Kings and allow a more consistent comparison, and a further improved YAG fine fibre could be achieved by removal of the porosity present in the sodium-free fibres.

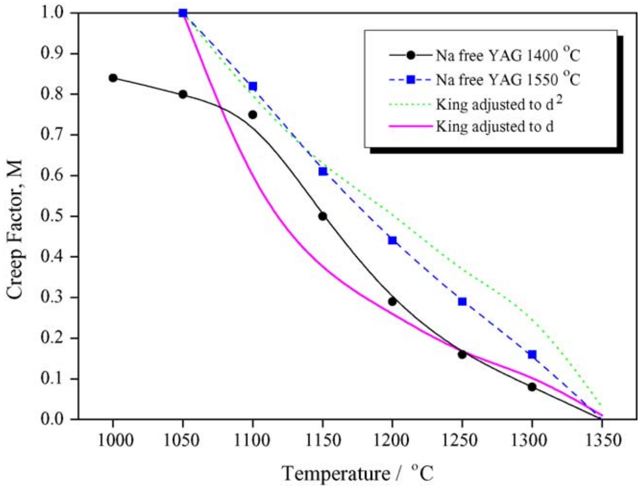

Fig. 8. Comparison of the of creep of sodium-free YAG fibres prefired to 1400 and 1550 °C/3 h against previously reported polycrystalline YAG fibres,[5] when adjusted for grain size, d, assuming: (a) Nabarro–Herring diffusion (strain $\propto d^2$) or (b) grain boundary effects (strain $\propto d$). As both processes will play a part, the true plot will lie somewhere between these two lines.

4. Conclusions

The use of a sodium-free aluminium chlorohydrate solution appeared to induce further structuring in the precursor sol, causing the sol to gel at relatively low concentrations and inducing viscoelasticity during spinning. However, high quality aligned fine fibres were nevertheless spun, and upon firing in air these formed pure phase YAG at 900 °C. When steamed over 200–500 °C for 3 h, the gel fibre formed single phase crystalline YAG between 400 and 500 °C, an extremely low temperature for this material to crystallise, 300 °C lower than in the fibres containing sodium. Upon postfiring to 1400 and 1550 °C, grains between 0.25 and 0.6 μm and pores between 0.08 and 0.17 μm had formed in the 4 μm diameter fibres. Despite this porosity, and smaller grain size when fired to 1550 °C, the sodium-free fibre exhibited superior creep resistance, creeping at temperatures 50 °C higher than the fibres previously made by the authors. This demonstrates that even small levels of sodium contamination are harmful to creep resistance in YAG.

References

1. Liu, Y., Zhang, Z., King, B., Halloran, J. and Laine, R. M., Synthesis of yttrium aluminum garnet from yttrium and aluminum isobutyrate precursors. *J. Am. Ceram. Soc.*, 1996, **79**, 385–394.
2. Corman, G. S., *Creep of Oxide Single Crystals. Final Report, September 1987–September 1989*. Government Research Announcements and Index, USA, 1990.
3. Karato, S., Wang, Z. and Fujino, K., High temperature creep of yttrium–aluminium garnet single crystals. *J. Mater. Sci.*, 1994, **29**, 6458–6462.
4. Parthasarathy, T. A., Mah, T. and Keller, K., High-temperature deformation behavior of polycrystalline yttrium aluminum garnet. *Ceram. Eng. Sci. Proc.*, 1991, **12**, 1767–1773.
5. King, B. H. and Halloran, J. W., Polycrystalline yttrium aluminum garnet fibers from colloidal sols. *J. Am. Ceram. Soc.*, 1995, **78**, 2141–2148.
6. Deng, S., High temperature creep behaviour of single crystal oxides. *J. Mater. Sci.*, 1996, **31**, 6077–6083.
7. Parthasarathy, T. A., Mah, T. and Keller, K., Creep mechanism of polycrystalline yttrium aluminum garnet. *J. Am. Ceram. Soc.*, 1992, **75**, 1756–1759.
8. Pullar, R. C., Taylor, M. D. and Bhattacharya, A. K., The manufacture of yttrium aluminium garnet (YAG) fibres by blow spinning from a sol–gel precursor. *J. Eur. Ceram. Soc.*, 1998, **18**, 1759–1764.
9. Morton, M. J., Birchall, J. D. and Cassidy, J. E., ICI, *UK Patent 1360200*, 1974.
10. Stacey, M. H. and Taylor, M. D., ICI, *European Patent 318203*, 1987.
11. Pullar, R. C., Taylor, M. D. and Bhattacharya, A. K., The sintering behaviour, mechanical properties and creep resistance of aligned polycrystalline yttrium aluminium garnet (YAG) fibres, produced from an aqueous sol–gel precursor. *J. Eur. Ceram. Soc.*, 1999, **19**, 1747–1758.
12. Pullar, R. C., Stacey, M. H., Taylor, M. D. and Bhattacharya, A. K., The decomposition, shrinkage and evolution with temperature of aligned hexagonal ferrite fibres. *Acta Mater.*, 2001, **49**, 4241–4250.
13. Pullar, R. C. and Bhattacharya, A. K., Polycrystalline yttrium aluminium garnet (YAG) fibres produced from the steaming of an aqueous sol–gel precursor. *Mater. Lett.*, 1999, **39**, 173–178.
14. Smith, M., Personal communication, ICI Saffil Business, Widnes, UK.
15. *Certificate of Analysis of Chlorohydrol Solution*. Reheis Inc., 1997.
16. Higgins, I. and Matthey, J., Personal communication, Widnes, UK.
17. Morscher, G. N. and Dicarlo, J. A., A simple test for thermomechanical evaluation of ceramic fibers. *J. Am. Ceram. Soc.*, 1992, **75**, 136–140.
18. King, B. H., Liu, Y., Laine, R. M. and Halloran, J. W., Fabrication of yttrium aluminate fibers. *Ceram. Eng. Sci. Proc.*, 1993, **14**, 639–650.
19. Liu, Y., Zhang, Z., King, B., Halloran, J. and Laine, R. M., Yttrium aluminum garnet fibers from metalloorganic precursors. *J. Am. Ceram. Soc.*, 1998, **81**, 629–645.