Eng. & Technology, Vol.24, No.3, 2005

High Temperature Creep Resistance Development Of Alumina Based Fibers By Heat Treatment

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Received on13/3/2004
Accepted on: 7/11/2004

Abstract

The behavior of alumina-oxide based fibers reinforcements for high temperature application composite materials. These fibers suffered from a process of grain growth fabrication process and that lead to a great change of the properties of fibers. To study these effects, we examine a commercially available (alumina) and (alumina-silica) fibers which heat treated at various temperature as annealing at 1100,1300 oC, for up to 100 hrs changes of the fiber tensile creep stress rupture with the microstructure changes together with the heat treatment was shown to be of a significant effect, especially on the alumina fiber and shows a good creep behavior (creep resistance).

الخلاصة

في هذا البحث تمت دراسة تطوير سلوك اوكسيد الالومينا اعتماداً على التقوية للالياف الاساسية حيث ان نمو حبيبات هذه الالياف تعتمد على المعاملات الحرارية وبالتالي الى تغير كبير في المواصفات الميكانيكية لهذه الالياف الاساسية . ولدراسة هذه التاثيرات تم فحص الالومينا وسليكات الالومينا المتيسرة محلياً والتي سوف يتم معالجتها حرارياً بدرجات حرارة مختلفة تصل الى (1100 و 1300 درجة مئوية) لاكثر من مائة ساعة . ان اختبار اجهاد شد كسر الزحف مع تغيرات التركيب المجهري وكذلك المعاملة الحرارية سوف تعطي تاثيراً واضحاً وخصوصاً على الياف الالومينا للحصول على سلوك زحف جيد أي مقاومة زحف جيدة عند درجات العالية .

Introduction

Earlier studies [1 - 3] on the alumina - based fibers have investigated that the mechanical properties of the fibers are depends on the history of material heat treatment. Remembering that, these fibers are considered as a reinforcements for the composite material for high temperature application so that, it is very important to develop an understanding "of the effects of heat treatment" during composite fabrication on such

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https://doi.org/10.30684/etj.24.3A.3
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fiber properties such as tensile stress, creep and stress rupture.

The recent studies on the same polycrystalline alumina fiber showed, that, the grain growth is very little at a temperature up to 1200 °C for a times of 10 hrs [2,3]. Also, these studies showed that, with free load conditions, the tensile strength are decreased due to the growth of critical flaws. But for the (alumina - silica) composite fiber, heat treatment above 1127 °C makes the amorphous silica to transform and the alumina mixture transform to mullet [1]. The as-produced alumina silica fibers tested at 1000 to 1200 °C [4], exhibits a great creep rates than did alumina in the same temperature and stress range [5]. However, no creep testing was performed on (alumin - silica) fibers after they were annealed to the mullet composition. Another (alumin-silica) fiber with a mullet-alumina composition was shown to have creep resistance greater than obtained by polycrystalline alumina fiber [1]. This means that, it is applicable to develop a commercially available alumina- silica fiber.

During the heat-treatment cycle, the instability in the microstructure of the alumina-silica fiber transform to a more stable.

The aim of this investigation was to determine the effects of heat treatment on the creep and rupture behavior of two commercially available oxide fibers, alumina and alumin-silica fiber, see table (1).

To make these results more useful, the creep behavior of these fibers is compared to other oxide fibers, including yttrium aluminum garnet fibers.

Experimental Procedure

The properties of as-produced properties are reported by their respective manufactures, since these materials were imported for special applications. The heat treatment processes includes annealing took place in an electric furnace and cooling in air over a (1 -100) hrs at 1100 °C and for 3 hrs at 1300 °C. The primary reason of choosing these temperatures is that they represent the upper temperatures for fabrication of ceramic-matrix composites

The tensile creep and rupture data were obtained by using a special apparatus as shown in figure (1) for tensile creep testing in air. These data are obtained between 900 and 1200 °C in air for times ranging

from 0.1 to 100 hrs and the applied stresses are of 190 ~ 280 Mpa. Individual fibers were glued to paper grips that were attached to hooks well out - side the hot zone (This zone is similar to that used in obtaining single crystal) except that it could reach 1500 °C.

The tensile creep deformation measurements were made at the lower hook using extensometer.

The measurements were taken every minute for the first half hour and then every half hour there after. The thermocouples used for temperature control and measurement respectively, were placed at the center of the hot zone, and for the creep - strain calculations the effective gauge length was taken to be 25 mm.

Results and Discussion

The tensile creep curves for the as-recieved alumina fibers at various stress levels are shown in figure (1) at 950 °C, and this figure shows the effects of the 1100 °C air anneal for 100 hrs. The curves exhibit a steady - state creep with little creep strain. The as received fiber, tested only at 275 Mpa, However, at the same stress, the annealed fiber displayed a much lower creep strain, just under 0.8 %. Moreover, the annealed fiber showed no indication of tertiary creep, even after more than 100 hrs but the stress on annealed fibers was increased to 413 Mpa, which resulted in an n increase of 3 strains after 50 hrs. When the creep temperature was raised to 1090 °C the strain in the annealed fiber exceeded 0.3 % within 2 hrs at 138 Mpa (Figure 2), while the as received fiber failed after only 10 min at the same stress. That means the doubling stress on the annealed fiber increased the creep strain by factor of 5.

The response of alumina - silica fiber creep response is illustrated in (Figure 3) at 1090 °C. Both samples the as received and the annealed samples at 1100 °C for 100 hrs were tested. The alumina - Silica required about 50 hrs instead of 2 hrs to achieve strain levels comparable to annealed alumina fiber (of figure 2).

The comparison between the annealed and as received fibers at 1090 °C with the same 138 Mpa load exhibits one order of magnitude less creep strain in the annealed fiber.

These results proved that the correct sequence of annealing process play an

important role in the creep behavior of these fibers. The effects of process annealing on the 0.2 % creep strength of the alumina fiber and alumina – silica fibers are shown in (figure 4 a, and b). Alumina fiber at 980 °C required (1 – 200 hrs) reaching 0.2 % creep at stresses from 700 to 100 Mpa. After the annealing process of the alumina fiber samples at 1100 °C for 100 hrs, the 0.2 % creep times increased from approximately (2 - 500 hrs) for the same stress, while at 1090 °C, there is no large annealing effect could be recognized in the as received alumina - fiber creep strength data. In contrast, the annealed alumina - silica fibers show a marked improvement over both the as received and annealed alumina fibers in the same stress range. Annealing of alumina - silica showed about an order of magnitude increasing in the time needed to reach 0.2 % strain at both 980 °C and 1090 °C. In .fact, the annealed fiber testing at 1090 °C behaved similar to the as received alumina - silica at 980 °C. The real stress - rapture data were limited, but (Figure 5) shows that always, annealing processes increased rupture time in comparison to the as received fibers. However, annealed alumina - silica shows only a slight increase in rupture time at 980 °C.

The micrographs of the alumina fibers in the as received and annealed condition (100 hrs at 1100 °C) shows that the initial average grain size of 0.1 μm appears unchanged. In contrast, the alumin - silica microstructure shown in (figure 6) shows that the initial 0.02 μm grain size increased four times after a 3 hrs, 1300 °C exposure, while alumina, the alumina - silica grain morphology remains equated after being exposed to this temperature.

Conclusions

1. The annealing process can be used to modify the high temperature creep deformation behavior of commercial polycrystalline - alumina – based fibers, and That make it is possible to improve the high temperature creep resistance of composites without affecting their stress - rupture behavior.
2. Annealing process produces phase and microstructure changes that allow them to become as creep resistant as yttrium aluminum.
3. The results of this work, show that the creep

properties of the as received fiber may not be appropriate for modeling composite creep behavior if the fiber creep properties are changed by fabrication or service conditions.

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Materials	Al_2O_3%	SiO_2%	Fe_2O_3%
Alumina based fiber	99	0.2	0.8

Table (1) Chemical composition of tested material

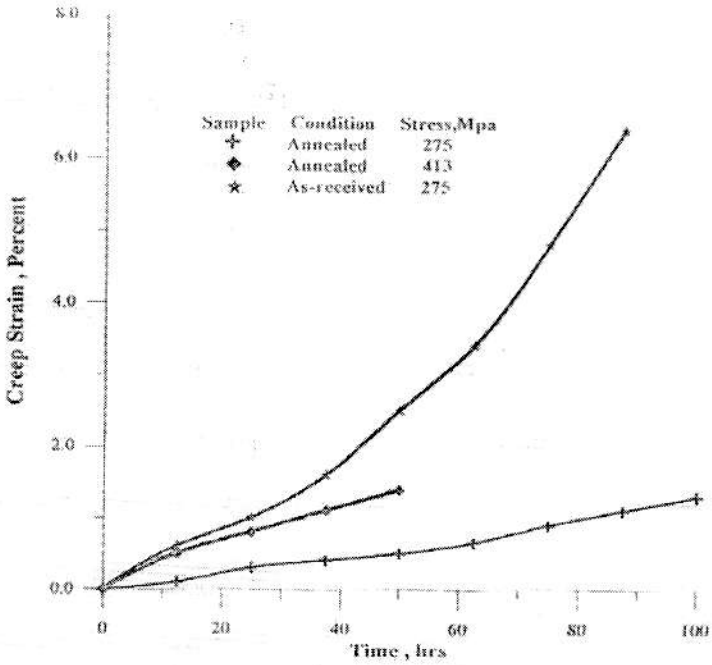

Figure (4) Typical tensile creep curves for as - received and Annealed Alumina - Fiber under stresses of 275 and 413 Mpa at 980 C (arrows indicate interrupted tests)

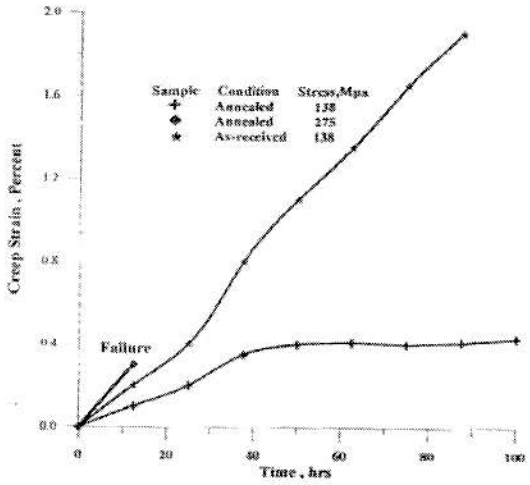

Figure (2) tensile creep curves for as - received and Annealed Alumina - Fiber at creep temperature of 1090 C (arrows indicate interrupted tests)

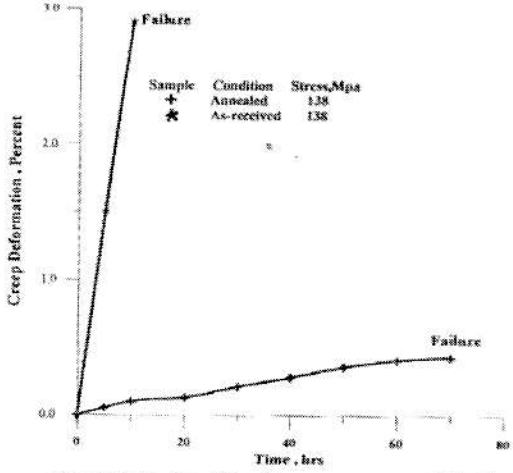

Figure (3) Alumina - Silica fibercreep response at 1090 C as a function of annealing at 1100 C for 100 hr

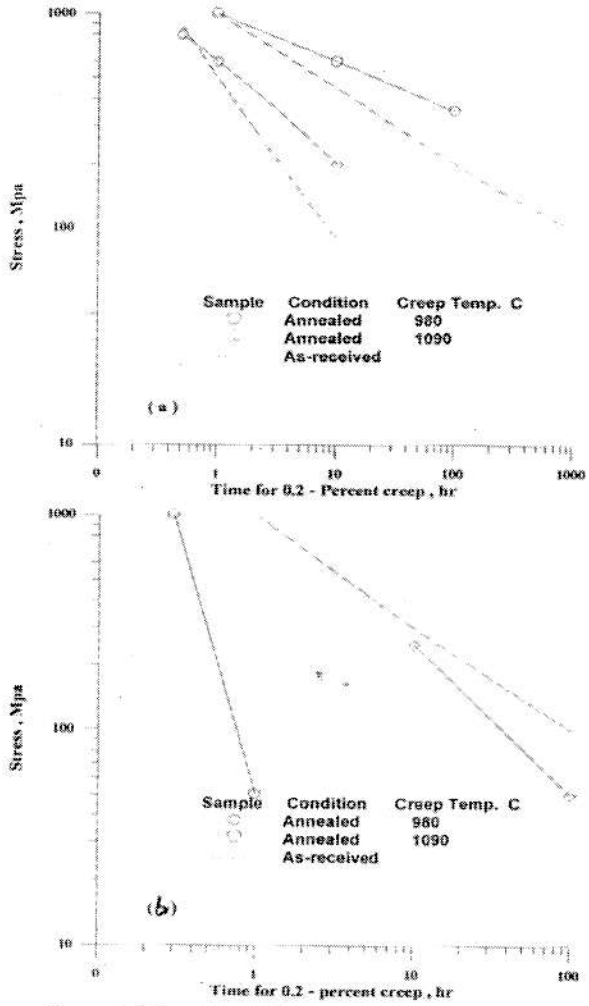

Figure (9) Effects of annealing on 0.2 - percent creep strength
(a) Alumina - fiber .(b) Alumina - Silica fiber.

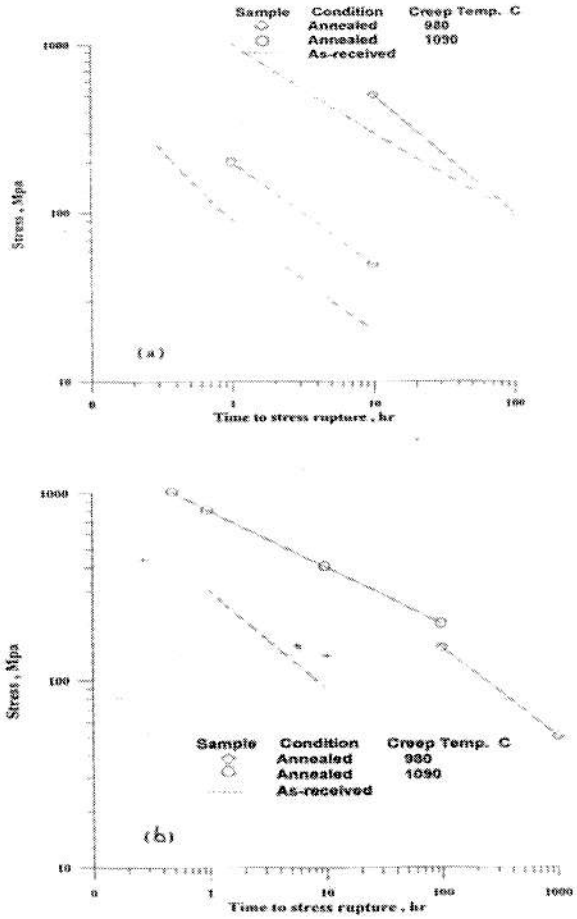

Figure (5) Annealing increases rupture time of as-received oxide fibers (arrows indicate interrupted tests) (a) Alumina - fiber .(b) Alumina - Silica fiber.

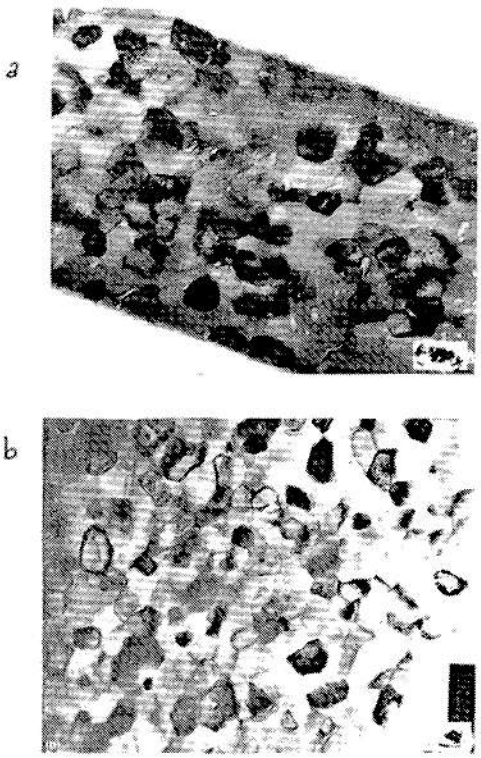

Figure (6) Alumina – silica fiber grain size increased fourfold after 3-hrs exposure at 1300 °C.
(a) As – received grain size = 0.2 μm (b) After annealing at 1300 oC.