EFFECTS OF HIGH TEMPERATURES ON ALUMINA CERAMIC FIBERS

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Abstract
Alumina ceramic fibers show a loss in tensile strength due to creep and grain growth at high temperatures. The diffences in the diffraction patterns of commercially available ceramic fibers are detected with wide angle x-ray diffraction. Therefor the fibers are exposed to different high temperatures and after that, the properties are examined. First of all, diffences betweent the diffent types of the fibers are shown. After that a comparison betweent fibers without and with a heat treatment at 1000 °C for 20 hours are made.

1. Introduction

Ceramic fibers are used in the production of ceramic matrix composites (CMC). CMCs are composed of ceramic material reinforced by ceramic fibers, thereby showing most of the positive properties of ceramics while being less brittle. They are mainly used in fields where a high-temperature resistance, a light weight or a high modulus are needed. [1-3]
The two general kinds of fibers are oxide and non-oxide ceramic fibers. The largest part of non-oxide ceramic fibers are based on SiC. Some of these fibers also contain nitrogen or boron. Their production is complex and expensive, since their production has to take place under non-oxidizing conditions. They have a high modulus and tensile strength as well as a low creep rate at high temperatures. Since those fibers are already quite well researched, the research focus moved to producing them at lower costs. [1,2,4]
The second kind of fiber, the oxide ceramic fibers, consist mainly of Al_2O_3 ceramics. While they show mostly the same or a littlem lower values concerning tensile strength and modulus as non-oxide fibers, they are less susceptible to damage through oxidation. The downside is that they have higher creep rates at high temperatures, which is why the focus of research is to lower this rates. [1,2]
Current manufacturers of oxide ceramic fibers are 3M, Cerafib and Nitivy. 3M™ introduced 1974 the first aluminum oxide based ceramic fiber and sells them under the name Nextel 312. This fiber is composed of 62 % Al_2O_3, 24 % SiO_2 and 14 % B_2O_3. [4,5] Then more fibers of the Nextel series were developed by with different amounts of silica or other additives such as boronoxide. [6]
Cerafib GmbH developed the fibers Cerafib 75 and Cerafib 99, which in the characteristics are similar to the fibers of the later Nextel series. The fiber Cerafib 75 consists of 75 % Al_2O_3 and 25 % SiO_2. The fiber Cerafib 99 consists of 99 % Al_2O_3 and 1 % oxide additives.
The developed oxide fiber NITIVY ALF of NITIVY Co., Ltd differs from other commercially available fibers, because a different crystal type of alumina is used. Instead of α-Al_2O_3 the γ-Al_2O_3 form was used. This fibers consist of 72 % Al_2O_3 and 28 % SiO_2. [7] Nevertheless, these fibers differ

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little in the properties from similar Nextel fibers such as Nextel 550. Despite the development of various fibers with different materials, the oxide fibers still have high creep rates at high temperatures.

2. Material and methods

In this study, commercial oxide ceramis fibers are examined. First of all, fibers from 3M, Cerafib and Nitivy (Nextel 610, Cerafib 75, Cerafib 99 and Nitivy Alf) are exposed to high temperatures (600 °C, 700 °C, 800 °C, 900 °C and 1000 °C) for 20 hours. In (Table 1) the different compositions of the fibers are shown.

Table 1. Compositions of the diffent fibers.

Name	Al_2O_3 (%)	SiO_2 (%)	*Other Oxides* (%)
Nextel 610	99	0	1
Cerafib 99	99	0	1
Cerafib 75	75	25	0
Nitivy Alf	72	28	0

After this treatment wide angle x-ray diffraction (WAXD) is used to detect diffences is the structure of the fibers caused by the high temeratures. The wide angle x-ray diffraction (WAXD) is carried out on the Image-Plate Detection System II, STOE & Cie GmbH, Darmstadt, Germany. Here, the resulting diffraction pattern is analysed. Peaks for the crystalline and amorphous fractions of the samples are adjusted. The areas of these peaks are evaluated and used to calculate the crystallinity X_c. It is obtained by the sum of the areas of the crystalline peaks A_x divided by the sum of all the peaks, shown in Eq. (1). A_a is the sum of the areas of the amorphous fractions.

$$X_c = \frac{A_x}{A_x + A_a} \tag{1}$$

Likewise, by differences in the width of the peaks conclusions on the grain size are made. So the effects of high temperatures on alumina fibers are examined.

3. Results and Discussion

Although the fibers Nextel 610 and Cerafib 99 are of the same composition there are differences in the diffraction patterns of fibers without heat treatment recognizable (Figure 1**).** The diffraction rings in (Figure 1a) appear very uniform. This is a sign, that there are no or just a few orientations in the fiber form cerafib. In contrast, there are orientations of the crystallite grains in the fiber from Nextel. This orientation increases with increasing heat treatment temperature.

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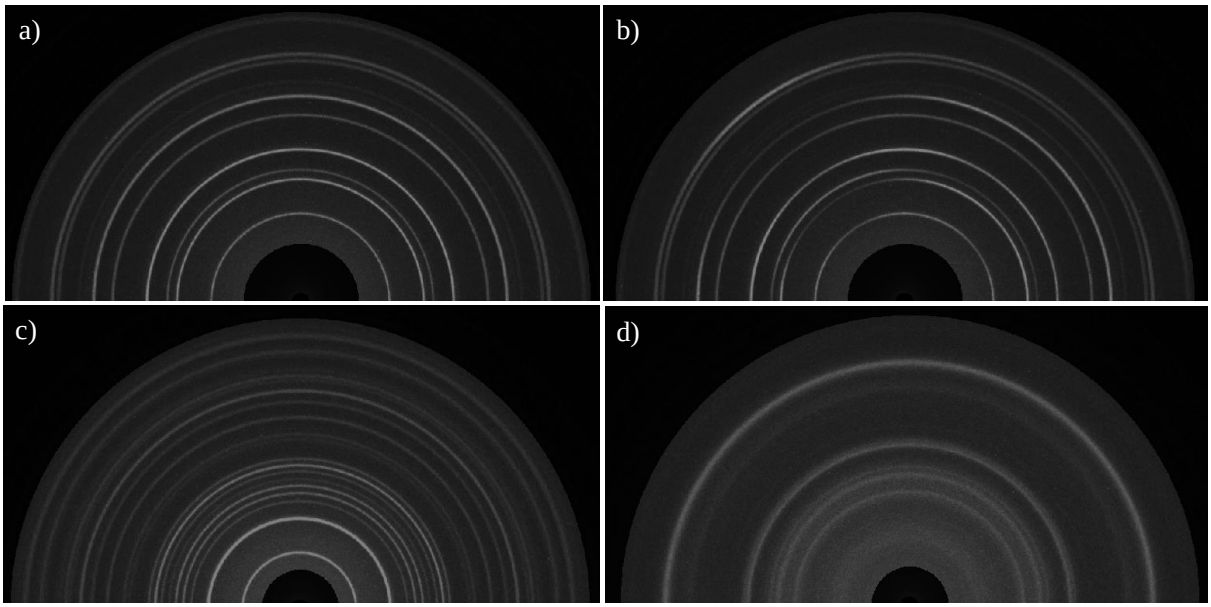

Figure 1. diffraction pattern of a) Cerafib 99 and b) Nextel 610 c) Cerafib 75 and d) Nitivy Alf fibers without heat treatment

In (Fig. 1c) are a lot more diffractions rings because of the SiO_2 in the fibers. Some of tis rings can be fond in (Fig. 1d), die diffraction pattern of Nitivy Alf fibers. But a very huge diffenence here is the crystalline phase of alumina and a big fraction of amorphous SiO_2. No diffences caused by the heat treatment are shown in this images, so they are converted into diagrams, that show the intensity above the diffraction angle 2θ (Figure 2). Here, only very small differences in the diagrams are recognizable, when each of the upper diagram is compared with the diagram underneath. The peaks apper sharper after the heat treatment. This is a sign for crystal growth into the small amorphous parts of the fibers and thus a higher crystallinity afterwards.
The calculations according to formula (1) show no significant increase in crystallinity. A trend can be seen, which is in the range of standard deviation of the measurement method, which is why it is neglected here.

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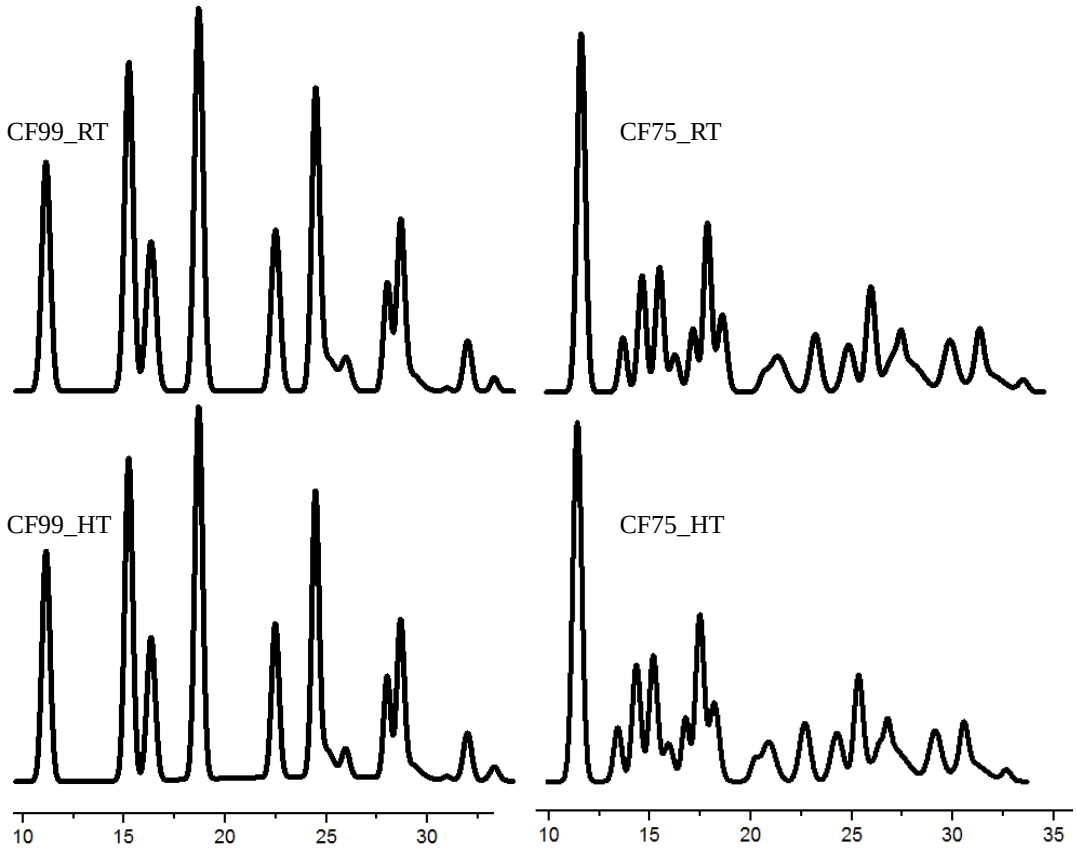

Figure 2. Intensity above diffraction angle 2θ from Cerafib 99 fibers (left) with (below, CF99_HT) and without (above, CF99_RT) heat treatment at 1000 °C and Cerafib 75 fibers (right) with (below, CF75_HT) and without (above, CF75_RT) heat treatment at 1000 °C

4. Conclusions

In ths Study the influence of high temeratures (up to 1000 °C) on the structre of commercial oxide ceramic fibers is detected. The structure is detected though wide angle x-ray diffraction. There a small change caused by the heat treatment is visible. To make further assumptions, mechanical tests are carried out on the samples in the future.

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