



# Experimental and numerical investigations on the tensile behavior of 3D random fibrous materials at elevated temperature



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## ABSTRACT

Using experimental method and Finite Element Method (FEM) in this paper, we studied the mechanical behaviors of the three-dimensional (3D) random fibrous (RF) material under tensile loadings in the through-the-thickness (TTT) and in-plane (IP) directions at elevated temperatures. It is interestingly found from the experiments that the fracture strength of 3D RF material in the TTT and IP directions doesn't follow the convention that the strength decreases as increasing temperature. Such a different variation trend of the fracture strength in the two directions is attributed to the presence of residual stress inside the material arisen from the fabrication process. In addition, Young's modulus of the 3D RF material in the TTT and IP directions decreases with the temperature. These observations from the experiment are in good agreement with the numerical results from the 3D FEM modeling of the 3D RF material developed in this paper.

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## 1. Introduction

Inorganic random fibrous (RF) materials with three-dimensional (3D) bonded networks are notable for their high porosity, low weight, high specific surface area and excellent thermal properties [1–6]. Due to these unique properties, 3D RF materials have been extensively used in a variety of fields, such as building, filtration and thermal insulation of space shuttle [3,4,7,8]. Applications to space shuttle usually impose the most stringent requirements on 3D RF materials due to severe thermal and mechanical service environments [5]. An early typical thermal protection system (TPS) of the space shuttle contains reusable surface insulation (RSI) as a kind of 3D RF materials, room-temperature vulcanizing (RTV) silica adhesive, and strain isolator pad (SIP) [9]. Numerous studies have been focused on the mechanical properties of TPS and the 3D RF materials at room temperature. It was found that the strength of the TPS system decreases with increasing loading cycles and shows the highly nonlinear performance [9–11] and the strength of 3D RF materials in the in-plane (IP) direction is stronger than that in the through-the-thickness (TTT) direction and increases as increasing the strain rate. However, the modulus of 3D RF materials is somewhat insensitive to the strain rate [12]. In addition, the mechanical properties of 3D RF materials are remarkably affected by the complex meso-

structures inside themselves, such as the structural randomness [13,14]. This prevents us from developing an accurate theoretical model to predict and simulate the mechanical properties of 3D RF materials. That is why the predictions of the theoretical models did not agree well with the experimental data [15].

Compared to theoretical models, Finite Element Method (FEM) modeling enables us to establish the relationship between macro-properties and meso-level details of RF materials [16–18]. Till now, numerous efforts have been invested to evaluate the mechanical properties of RF materials using FEM modeling. These studies include the onset and propagation of damage in the fiber and bonding points and the effects of orientation distribution function from low-density RF materials [19]. Moreover, some modeling results such as the tensile performance and damage micro-mechanisms of glass fiber non-woven felts (a kind of RF materials) has been found to agree well with the experimental counterparts [20]. These studies are mainly based on two-dimensional (2D) model. Other examples of 2D model based simulations have been applied to study the elastic-plastic behavior of porous metal fiber-sintered sheets and flexible fiber-fiber bonding [21–24]. A further advance in FEM modeling was made by Lu et al. [3,4]. They established a 3D FEM model to explore the tensile and compressive performance of 3D RF materials. This model was successfully used to mimic the morphology of 3D RF materials observed in the experiments. Their simulation results were in good agreement with the experimental results at room temperature.

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Up to now, although numerous efforts have been invested into the study of 3D RF materials, most of studies were only carried out at the room temperature. Thus, we may raise a question, i.e., how does the mechanical behaviors of 3D RF materials change at elevated temperature? In this paper, we will attempt to answer it using experimental method and FEM modeling. In this study, we firstly performed the tensile tests of 3D RF material in the two directions, i.e., TTT and IP, at the elevated temperatures ranging from 299 K to 1273 K. The tensile response of 3D RF material in the two directions, as well as the fracture strength and Young's modulus at the different temperatures are obtained. To reveal the material properties dependent on the temperature, a 3D FEM model due to Lu et al. [3,4] is adopted in this study. We further extend this 3D FEM model by incorporating the temperature effects and residual stress into it, so that it is capable of simulating the mechanical behavior of 3D RF material at elevated temperatures. Finally, we compare the results obtained from experiments and FEM simulation. It is found that they are in good agreement.

## 2. Experiment

### 2.1. Materials

In this study, the 3D RF material was sintered by high purity silica fibers (purity  $\geq 99.95\%$ ) and mullite fibers. The process for sintering are detailed in Ref. [3] and a brief introduction is only made here. Firstly, the fibers are cut into short fibers with averaging length 0.6 mm and diameter 0.01 mm, then the short fibers are mixed with the sintering additive ( $B_4C$  power and soluble starch) to produce the slurry. The slurry is poured into a mold and the water is evaporated in the vacuum. After sufficient drying, the fiber body is sintered in the furnace at 1473 K for 2 h. During the sintering process, the randomly distributed fibers are bonded together. Finally, the highly porous fiber networks are constructed. After cooling down to the room temperature in the furnace, the 3D RF material is fabricated. Fig. 1a and b show the scanning electron

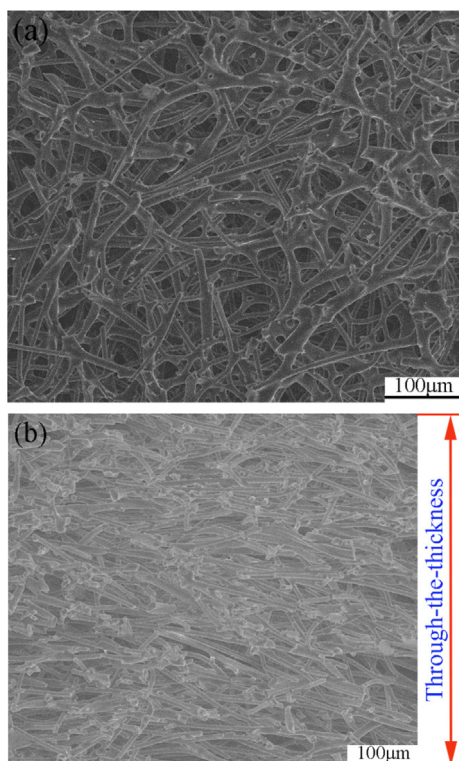


Fig. 1. SEM images of the 3D RF material in the (a) TTT and (b) IP directions.

microscope (SEM) images of the 3D RF material in the TTT and IP directions respectively. Clearly, the fibers distribute randomly and have high porosity in the TTT direction (Fig. 1a). However, they exhibit the preferred orientation in the IP direction (Fig. 1b). The averaging density and porosity of the 3D RF material are  $0.33 \text{ g/cm}^3$  and 83%, respectively.

### 2.2. Experiment devices

The tensile tests for specimens are conducted on a Universal Test Machine (Z005, Zwick Roell, Germany) and a 1473 K high temperature mechanics test system (GW 1200, Changchun Fangrui Technology CO. LTD, China) at the loading rate of 1 mm/min through the displacement control model. During the testing, the furnace is held for 20 min at the testing temperature (except room temperature) before the tensile tests are performed. A high temperature furnace extensometer (Model 3448, Epsilon Technology Corp, USA) is used to measure the displacement of the specimen.

### 2.3. Tension specimen and fixture

Fig. 2a plots the shape and dimensions of the tensile specimen. Two groups of specimens are cut out of 3D RF bulk material in the TTT and IP directions. To fix the both ends of specimen in the furnace, we designed a fixture as schematically shown in Fig. 2b. The specimen and fixture are then assembled in the furnace as displayed in Fig. 2c. With such a testing system, we could perform the tensile test at an elevated temperature up to 1373 K.

### 2.4. Experimental results

It will be seen that the random distribution of fibers in the 3D RF material imparts the variations in the mechanical response of 3D RF material during the tensile testing. Here, only some typical load-displacement curves in the TTT and IP directions at the different temperatures are shown in Fig. 3. On the whole, the 3D RF material in the TTT and IP directions exhibit the brittle fracture when the temperature is below 1073 K. However, it shows the ductile fracture as temperature is 1273 K.

Further inspection of loading curves reveals that fracture strength of 3D RF material in the TTT and IP directions doesn't follow the convention that the strength decreases as increasing temperature. For example, the strength at 1073 K is higher than those at 573 K and 773 K in the TTT direction (Fig. 3a). Moreover, no clear variation trend of the fracture strength can be seen in the IP direction (Fig. 3b). To better reveal the variation trends of fracture strengths in the TTT and IP directions, we further plotted them vs. temperature in Fig. 4. Clearly, the fracture strength in the IP direction is almost six times greater than that in the TTT direction. Such differences can be attributed to the different distribution of fibers in two directions. This is mainly due to the preferred orientation of fibers in the IP direction (Fig. 1b), which can bear the load. The temperature-dependent trend of the strength will be further discussed in the Section 4. Fig. 5 displays the fracture morphology of specimens tested at 1273 K. We can see that the specimens fracture through breaking the fibers (the directions of arrows), while almost all the bonding zones keep intact.

## 3. FEM model

### 3.1. Element analysis of the material

Before proceeding to the FEM model setup, we need to analyze the material that bonds the two types of fibers in the 3D RF material. Fig. 6a shows a SEM image of local structure with some

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