Heat resistance study of basalt fiber material via mechanical tests

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Abstract. This paper focuses on the study of the relationship between the fracture strength of basalt rovings and temperature. Strong stretching performance of the rovings has been tested after the treatment at fixed temperatures but different heating time and then the fracture strength of the rovings exposed to the heating at different temperatures and cooled in different modes investigated. Finally, the fracture strength of the basalt material after the heat treatment was studied. The results showed that the room-temperature strength tends to decrease with an increase of the heat treatment time at 250°C, but it has the local maximum after 2h heating. And the basalt rovings strength increased after the heat treatment up to 200°C. It was 16.7 percent higher than the original strength. The strength depends not only on the temperature and duration of the heating, but also on the cooling mode. The value of the strength measured after cold water cooling was less by 6.3% compared with an ambient air cooling mode. The room-temperature breaking strength of the rovings heated at 200°C and 100°C for 2 hours each increased by about 14.6% with respect to unpretreated basalt rovings.

1. Introduction

With the development of industrial science and technology, the new fiber materials of high performance emerge, and the range of their applications expands. Among them basalt fiber materials are well known due to very high strength and excellent high temperature resistance. The studies of thermodynamic properties of basalt and glass fibers performed by Zhou [1] have shown that due to Fe₂O₃ and FeO compounds in the basalt yarn the working temperature of the basalt fiber can reach as high as 600°C, while glass fibers lose their strength at heating above 400°C. The morphology and thermal stability of the basalt and glass fibers were analyzed and compared by Hao et al [2]. Using the projection microscope and scanning electron microscopy, they found similar structure of the two types of fibers, but the thermal stability of basalt fibers is higher. After study the high temperature resistance of the basalt fibers Li [3] concluded that at 200°C they possess the best mechanical properties. Generally, the heat resistance of basalt fibers is not well investigated. Nevertheless, basalt fiber products are extensively used at high temperature in many industrial applications, and their functional temperature and heating time are different in different industrial sectors. In this study, we designed and constructed a testing system for the fracture strength measurements of the basalt fiber materials after their heat treatment at various temperatures, duration of heat exposure and different cooling modes, in order to find out the relationship between the mechanical
properties and parameters of the heat treatment of basalt fiber materials. The results obtained by this method will be used for the development of basalt fiber products and their practical applications.

2. Test materials, instruments, and test conditions

2.1 Test materials
264tex basalt non-twist roving, single fiber diameter 13 μm, fracture strength of 41.51 cN/Tex, provided by Sichuan Tuxin Basalt co., Ltd. (China).

2.2 Equipment and instruments
YG-01 electrical thermostatic drying oven (temperature control range of 0-400°C) was produced by the Tianjin city Test Instrument Co., Ltd. (China)
UTM5305 electronic universal power machine with WGDY - 7350L high-temperature box; Shenzhen established company
Test reference standard: GB/t25045-2010
Test conditions: temperature 20± 2°C, the relative humidity 64%.

3. Experimental study

3.1 Study of the relationship between the heating time and the material fracture strength
We made a series of 6 cm long samples of basalt rovings and put them in the electrical thermostatic drying oven, set the oven temperature 250°C, and the processed samples in the drying box for 0.5 h, 1 h, 1.5 h, 2h, 2.5 h, 3h, 3.5 h and 4h, then placed them out. After cooling, the tensile fracture strength of each specimen was tested and presented in table 1, 10 samples per group, then we calculated the average and plotted the fracture strength of the basalt roving as a function of the heating time at T=250 °C as shown in figure 1.

| Time(h) | Mean breaking strength(cN/tex) | Variation(%) |
|---------|-------------------------------|--------------|
| 0.5     | 39.1                          | 9.8          |
| 1.0     | 35.9                          | 9.0          |
| 1.5     | 38.5                          | 5.7          |
| 2.0     | 42.9                          | 7.0          |
| 2.5     | 39.3                          | 5.5          |
| 3.0     | 32.8                          | 10.3         |
| 3.5     | 31.5                          | 7.5          |
| 4.0     | 28.6                          | 9.3          |

As shown in figure 1, after the heating in the oven at a temperature of 250°C, the fracture strength of the basalt rovings changed with the change of the heating time. There was a certain decrease after the first hour treatment, then the breaking strength increased after heating up to 2 hours when the strength reached the maximum, but then it dropped again.

According to figure 1, we suppose that a strength loss in the initial stage of heating took place due to mass agglomeration of the surface treatment agent and the decrease of the cohesive force. The fracture strength increased after 1 hour heating probably due to better consolidation of the fibers as a result of volatilization of small molecules like as water and solvents, and the fiber surface became rougher. And the fracture strength decreased almost linearly after 2 hours heating, because the surface treatment agent volatilized greatly, and the
fiber structure, interatomic chemical bonds destroyed. After 4 hours heating, the strength of the rovings decreased by 31.6%.

Nevertheless, after basalt rovings heating for 3 hours at 250°C temperature their strength still was equal to about 80% of the original one. So far, the basalt fibers heat resistance is quite good.

3.2 Analysis of the fracture mechanism of the basalt fiber material after the heating
The cross-section electron microscopy of the basalt fiber bundles is shown in figure 2. A photo of the short stretch break of basalt roving is shown in figure 3.

Figure 2 implies that basalt fibers differ from conventional fibers or the other organic high performance fibers. The fracture is very smooth, the texture is hard, the same as a stone material. So after the basalt stone was processed into the fibers they are still a very brittle material. It can be seen from figure 3 that the basalt roving is broken; the beam fracture appears like an explosion. At drawing the monofilament elongation is small but the energy accumulation of a tension is high, after approaching the fiber strength limit the tensile force exceeds the limit of each single filament, all of the filaments will break simultaneously and form an explosive fracture, which is very different from the traditional way of breaking a yarn.

Figure 4 shows the displacement curve of the basaltic roving at the loading. As we can see from the curve, the displacement is very small until the fracture, and no more than 3 mm after the strength reaches the maximum and it falls off as a cliff. This means that the monofilaments were fractured almost simultaneously when the breaking strength reached the maximum. The load decreased slowly at initial stage, probably due to entanglement between fibers resulting in friction.
3.3 Relationship between the material fracture strength and the temperature

According to the described above sample preparation method, we prepared a series of samples at -20, -10, 0, 50, 100, 150, 200, 250, and 300 °C during 2 hours.

| Temperature(℃) | -20  | -10  |  0  |  50  | 100  | 150  | 200  | 250  | 300  |
|----------------|------|------|-----|------|------|------|------|------|------|
| Natural        | Mean breaking strength(cN/tex) | 43.6 | 45.1 | 44.2 | 43.6 | 45.3 | 46.8 | 48.5 | 42.6 | 26.9 |
|                | Variation (%)                  | 5.5  | 10.9 | 11.1 | 6.1  | 7.3  | 8.2  | 7.3  | 8.7  | 6.8  |
| Water cooled   | Mean breaking strength(cN/tex) | 42.5 | 42.8 | 38.5 | 39.5 | 40.6 | 42.7 | 40.9 | 40.1 | 30.8 |
|                | Variation (%)                  | 2.0  | 4.0  | 4.0  | 7.8  | 4.7  | 9.4  | 9.1  | 9.4  | 14.3 |

Table 2. Breaking strength of the basalt fiber under different heating temperature
Then they were dealt with in two different ways: one series was cooled quickly in cold water and the other one series was cooled at ambient conditions. Then the relationship between the temperature of the treatment and the room-temperature fracture strength were tested and the results are shown in table 2 and figure 5.

![Graph showing the relationship between temperature and breaking strength.](image)

**Figure 5.** The room-temperature fracture strength of the basalt rovings as a function of the temperature of the treatment

As the natural cooling curve in figure 5 shows, the fracture strength of the basalt roving materials increased gradually with the increase of the processing temperature up to 200°C. The breaking strength after the treatment at 200°C reaches the maximum value, which is higher than that of untreated material by 11.1%. Then in the range between 250 - 300 °C, the strength reduced by 44.4% referring to the highest value. The reason of a sustained decline in the fracture strength of basalt rovings is a change in the internal structure of the fibers and the volatilization of the surface treatment agents at the heating. Ying *et al* [4] have shown that basalt fiber can still maintain about 70% of the initial strength at 400°C. This may take place due to different basalt composition and the surface treatment agent.

As is seen in figure 5, the test results are different with different cooling ways after heating. The fracture strength of the basalt rovings cooled by cold water are generally less than the strength of the ones cooled at room temperature, excluding the case of the heating at 300 °C. The difference of the test values between the two different processing methods is about 16.3% for the samples heated at 200 °C.

As seen in figure 5, the strength of the basalt rovings cooled by cold water may be higher than those cooled at ambient conditions if samples were treated below -20°C. However, the low temperature range of the experimental equipment was limited, so the low-temperature range needs further study in the future.

3.4 Study of the strength retention of materials after heat treatment

The results described above show that the breaking strength reached the maximum increase of 16.7% after the heating at 200°C for 2 hours. In order to study the retainability of the strength gain, the following tests were carried out. Firstly, the basalt rovings were placed for 2 hours in
a 200°C environment. Then after the cooling, some of them were heated again for 2 hours at 50°C, 100°C, 150°C and 200°C. Finally, after cooling at room temperature they were tested and two curves of the breaking strength were plotted in figure 6 according to the results presented in table 3.

| Table 3. Breaking strength of the basalt fiber after pretreatment and without pretreatment |
|---------------------------------|----------------|----------------|----------------|----------------|
| Temperature(℃)                  | 50             | 100            | 150            | 200            |
| No pretreatment                 | Mean breaking strength(cN/tex) | 43.6           | 45.3           | 46.8           | 48.5           |
|                                | Variation (%)  | 6.1            | 7.3            | 8.2            | 7.3            |
| Treated at 200 °C in advance    | Mean breaking strength(cN/tex) | 45.9           | 52.2           | 46.2           | 43.1           |
|                                | Variation (%)  | 4.9            | 7.2            | 8.1            | 6.8            |

As is seen in figure 6, the fracture strength of the pretreated samples increased firstly and then decreased after reaching the maximum value at 100 °C. In this particular case the strength improved by about 14.6% with respect to unpretreated rovings. However, after the repeated treatment at 200°C the fracture strength decreased by 6.9%, as compared to the unpretreated basalt rovings. Nevertheless, basalt rovings heated two times at 200 °C, 2 hours each, still possess 93.1 percent the breaking strength of the original samples. This makes basalt fibers and rovings more suitable for the development of new temperature-resistant high-strength industrial materials than other raw materials.

![Figure 6. Comparison of the strength variation curves after two different treatment types](image)

4. Conclusions
We made the thermal testing system on the basis of UTM5305 electronic universal testing machine and electrical drying oven and used it for the studies of heating effect on basalt
rovings fracture strength. The mechanical tests have been carried out at room temperature after basalt rovings treatment at different temperatures with different heating time and different cooling modes. The following main conclusions are drawn from the research and analysis:

1. Basalt fiber material has a very good high temperature resistance, after three hours temperature processing at 250°C, the fracture strength still maintain 78.9% of the initial value.

2. The strength of the basalt fiber varies with the processing temperature, its fracture strength reaches the maximum after treatment at 200°C, it exceeds the initial one by 16.7%.

3. The average fracture strength of the basalt fiber rovings cooled by cold water after the heat treatment decreased on about 6.3% comparing with unprocessed sample.

4. The fracture strength of the basalt fiber rovings treated at 200°C temperature in advance, increased again after the next heat treatment up to 100 °C, its fracture strength increased by 14.63% comparing to the samples which were not pretreated at 200 °C temperature.

References

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