# STUDY ON PERFORMANCE OF DIATOMITE/BASALT FIBER COMPOSITE MODIFIED ASPHALT UNDER THE CONDITION OF ENERGY SAVING, GREEN AND LOW CARBON

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*In order to obtain composite modified asphalt with excellent performance of diatomite and basalt fiber suitable for the humid and hot areas of southern China, meet the requirements of carbon neutralization and carbon peak, and reduce energy consumption, the response surface methodology was taken to perfect the preparation process. The conventional physical properties of composite modified asphalt prepared in divergent basalt fibers and diatomite dosages and shear time conditions were tested separately, such as the penetration, softening point, melleability and viscosity, the optimal level values of each factor were obtained, and the high and low temperature performance of diatomite and basalt fiber modified bitumen at different dosages were evaluated by DSR and BBR tests. The test results show that the levels of various factors in the preparation process were comprehensively optimized by response surface methodology. When diatomite content is 11.18%, basalt fiber content is 3.09%, at 160°C, using 4700r/min rate of high-speed shear 44.2min, the composite modified asphalt has the best performance which is superior to that of SBS modified asphalt, while its low-temperature performance is slightly inferior to that of SBS modified asphalt.*

**Keywords:** energy saving, diatomite, basalt fiber, composite modification, response surface methodology, preparation process.

## 1. Introduction

In order to further improve the ability of bitumen pavement in hot and rainy climate and heavy load traffic, domestic and foreign relevant researches on composite modified asphalt materials have been carried out [[1]-[4]]. Currently, the modified asphalt is mainly polymer modified, but the polymer modifier is expensive, and the modification technology, equipment and construction process are relatively complex [5]. Meeting the requirements of energy-saving, green and

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low-carbon transportation infrastructure, the possibility of substituting polymer modifier for diatomite and basalt fiber was studied in this paper [6]. Diatomaceous earth is a natural siliceous sedimentary rock, and its main components are $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO and so on [7]. Diatomaceous earth owns the qualities of light weight, large surface area, high porosity, strong adsorption, sound insulation, and no chemical reaction with asphalt [8]. Studies have shown that adding diatomite into base asphalt is able to boost stability in high temperature, stability in water and asphalt's resistence to fatigue [9]-[13]. basalt fiber and asphalt binder and formed in asphalt space mesh structure, promote the stress dispersion and increase the elasticity, has obvious change resistance, crack resistance effect, and could enhance the road performance of bitumen pavement [14]-[15], specifically in the low temperature crack resistance. At present, there are few reports on the anti-shear performance, anti-crack performance, comprehensive performance evaluation and improvement mechanism of the slurry [16]-[17].

Given the current research status in these two types of bitumens in domestic and foreign [18], the preparation process needs to be optimized. Considering the key preparation parameters in the process of preparation, three influencing factors, including diatomite content, basalt fiber content and shear time, were selected, response surface method was used to analyze the effects of a variety of factors on the three indexes and viscosity of diatomite-basalt fiber compound modified pitch, and to determine the best preparation process of diatomite-basalt fiber compound modified asphalt. On this basis, the high temperature deformation and low temperature cracking resistance of diatomite basalt fiber compound modified pitch were studied and evaluated by shear rheological in dynamic and low temperature bending tests.

## 2. Materials and Methods

### 2.1 Asphalt

*Table 1*

**Technical Performance of Base Pitch Jinling Petrochemical–70#**

| Property | Technical Criterion | Measurement |
|---|---|---|
| Penetration (25℃, 0.1mm) | 60～80 | 63.2 |
| Softening temperature (℃) | ≥46 | 47.4 |
| Melleability (15℃, cm) | ≥100 | >100 |

*Table 2*

**Technical Properties of SK SBS (I-D) Modified Asphalt**

| Property | Technical Criterion | Measurement |
|---|---|---|
| Penetration (25℃, 0.1mm) | 40～60 | 52 |
| Melleability (5℃, cm) | ≥20 | 28 |
| Softening temperature (℃) | ≥76 | 78 |

Jinling Petrochemical 70# petroleum asphalt and control group SK SBS (I-D) modified asphalt with 4.5% SBS are selected to study. Their technical indexs are as followed in Table 1 and Table 2. The performance of bitumen conforms to the requirements of JTGF40-2004.

### 2.2 Diatomite

High quality diatomite, a siliceous sedimentary rock of biological origin, was selected from Perpetually White Mountain in Jilin Province. The chemical composition of diatomite is showed as followed.

*Table 3*

**Chemical Composition of Diatomite**

| Chemical Composition | $K_2O$ | MgO | $TiO_2$ | CaO | $Al_2O_3$ | $Fe_2O_3$ | $SiO_2$ | Loss on ignition |
|---|---|---|---|---|---|---|---|---|
| Proportion (%) | 0.67 | 0.45 | 0.3 | 0.52 | 4.5 | 1.5 | 85.6 | 1.61 |

### 2.3 Basalt Fiber

The basalt fiber specialized in the experiment was produced by Zhengzhou Dengdian Basalt Fiber Company Ltd. Its elementary properties are summed up in Table 4.

*Table 4*

**The Elementary Properties of Basalt Fiber**

| Index | Diameter (μm) | Length (mm) | Specific Gravity ($g/cm^3$) | linear density (tex) | Elongation at Break (%) | elastic modulus (MPa) | Tensile Strength (MPa) |
|---|---|---|---|---|---|---|---|
| Values | 17 | 6.11 | 2.679 | 235 | 1.97 | 75800 | 1450 |
| Requirements | / | / | 2.6-2.8 | / | ≤3.1 | ≥7500 | ≥1200 |

## 3. Test Method and Design

The details of response surface method design are as follows:

(1) Formulate a test scheme.

(2) Statistical and collate test data.

(3) The function relation between the argument and the response value is fitted by using multivariate quadratic regression equation.

(4) Through analyzing the regression equation, the optimal process argument are calculated and the multivariable problem is finally solved.

### 3.1 Use Response Surface Method to Determine the Optimal Dosage

#### 3.1.1 Preparation Process and Test Results

In this experiment, basalt fiber, base asphalt and diatomite with preset mixing amount were placed together in an oven at 140℃ for 3h. Box-Behnken

Design of corresponding surface method was adopted, 2%, 3%, 4% basalt fiber and 5%, 10%, 15% diatomite (two additives refer to the percentage of asphalt quality) were mixed in asphalt, the high speed shear instrument which can control the temperature with oil bath was used for shear mixing. The instrument was operated to control the two parameters of 160℃ and 4700r/min, stir for 30, 45 and 60 min to ensure that basalt fiber and diatomite were evenly distributed in asphalt, and the specimen should be poured immediately.

### 3.1.2 Separate Modification

According to the above preparation process, 5%, 10%, 15% diatomite and 2%, 3%, 4% basalt fibers were prepared respectively for routine three index and viscosity tests, as summarized in Table 5:

*Table 5*

**Three major indexes and viscosity of asphalt under single dosage**

| dosage | Penetration 25℃, 100g, 5s (0.1 mm) | Softening point (℃) | Ductility 5℃, 1cm/min (mm) | viscosity, 135℃ (Pa·s) |
|---|---|---|---|---|
| 0 | 63.2 | 47.4 | 91.2 | 0.48 |
| 5% diatomite | 57.8 | 52.5 | 76.3 | 0.52 |
| 10% | 53.2 | 54.6 | 72.6 | 0.61 |
| 15% diatomite | 51.3 | 55.7 | 70.2 | 0.73 |
| 2% basalt fiber | 51.5 | 52.4 | 54.7 | 0.50 |
| 3% basalt fiber | 50.2 | 54.3 | 53.1 | 0.54 |
| 4% basalt fiber | 49.3 | 53.5 | 50.1 | 0.58 |

### 3.1.3 Compound Modification

The design variables were A (content in diatomaceous earth), B (content in basalt fiber), and C (shear time). The response indexes were the penetration (25℃, 100g, 5 s)/ 0.1mm, softening point (°C), Meleability (5℃, 1cm/min)/mm and Viscosity. A three-factor +four-level composite experimental design was carried out in Box-Behnken (BBD) mode, in order to obtain the maximum dosage of diatomaceous earth and basalt fiber modification effect. The experimental data are summed up as followed.

*Table 6*

**Experimental design and results**

| Test number | A:diatomite (%) | B: basalt fiber(%) | C:shear time(min) | the penetration 25℃,(0.1 mm) | softening temperature(℃) | Melleability 5℃,(mm) | Viscosity 135℃, (Pa·s) |
|---|---|---|---|---|---|---|---|
| 1 | 5 | 3 | 60 | 52.7 | 55.3 | 61.2 | 0.69 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 2 | 5 | 2 | 45 | 49.7 | 52.8 | 64.1 | 0.79 |
| 3 | 5 | 3 | 30 | 46.6 | 56.3 | 59.7 | 0.74 |
| 4 | 5 | 4 | 45 | 56.9 | 55.8 | 55.3 | 0.55 |
| 5 | 10 | 3 | 45 | 47.8 | 59.1 | 57.7 | 0.76 |
| 6 | 10 | 4 | 30 | 48.2 | 58.3 | 54.8 | 0.80 |
| 7 | 10 | 4 | 60 | 48.2 | 57.2 | 54.2 | 0.76 |
| 8 | 10 | 3 | 45 | 46 | 59.3 | 58.6 | 0.73 |
| 9 | 10 | 3 | 45 | 46.5 | 59.6 | 58.5 | 0.74 |
| 10 | 10 | 2 | 60 | 48.1 | 56.4 | 59.6 | 0.73 |
| 11 | 10 | 3 | 45 | 45.9 | 58.9 | 58.4 | 0.72 |
| 12 | 10 | 3 | 45 | 46.9 | 59.1 | 57.9 | 0.73 |
| 13 | 10 | 2 | 30 | 52.4 | 56.1 | 58.9 | 0.68 |
| 14 | 15 | 2 | 45 | 49.8 | 57.3 | 56.4 | 0.71 |
| 15 | 15 | 3 | 30 | 48.9 | 57.1 | 54.8 | 0.72 |
| 16 | 15 | 3 | 60 | 47.4 | 57.6 | 54.6 | 0.81 |
| 17 | 15 | 4 | 45 | 48.6 | 59.2 | 53.7 | 0.85 |

### 3.2 Validity Analysis

*Table 7*

**Penetration Analysis of Between-Subjects Effects Table**

| Source | quadratic sum | df | Mean Square | F-Value | P-Value | |
|---|---|---|---|---|---|---|
| Model | 102.91 | 9 | 11.43 | 19.55 | 0.0004 | Significant |
| A-diatomite | 37.41 | 1 | 37.41 | 63.96 | <0.0001 | |
| B-basalt fiber | 38.28 | 1 | 38.28 | 65.45 | <0.0001 | |
| C-shear time | 2.64 | 1 | 2.64 | 4.52 | 0.0710 | |
| AB | 1.69 | 1 | 1.69 | 2.89 | 0.1330 | |
| AC | 0.3025 | 1 | 0.3025 | 0.5172 | 0.4954 | |
| BC | 0.3025 | 1 | 0.3025 | 0.5172 | 0.4954 | |
| $A^2$ | 16.51 | 1 | 16.51 | 28.22 | 0.0011 | |
| $B^2$ | 0.0017 | 1 | 0.0017 | 0.0029 | 0.9587 | |
| $C^2$ | 4.69 | 1 | 4.69 | 8.01 | 0.0254 | |

The data results obtained according to the design scheme are analyzed in combination with Table 6. Selecting the penetration P as the research object, and the quadratic regression equation was simulated by Design-Expert software and response surface method, the significance of simulation parameters was verified by drawing diagrams. Analysis of variance for penetration is shown in Table 7.

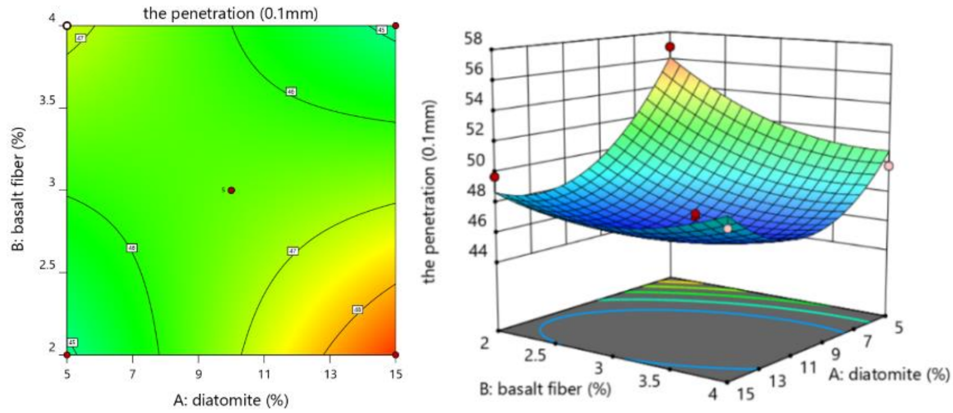
The penetration of the response index adopts a quadratic polynomial fitting equation. The fitted equation of the response exponent is obtained as:

$$Y=46.38-2.24A-1.15B-0.1125C+1.5AB-0.275AC+0.3BC+3.4A^2+1.47B^2+0.3975C^2$$

Where A is the amount of diatomite; B is basalt fiber content; C is the shear time.

The reliability analysis of the expression of equation Y shows that the $R^2$ of equation Y is 0.95, which verifies that BBD model is effective to predict the change of penetration degree.

### 3.3 Response Surface Model Analysis




(a) (b)

Fig. 1. Contour plot and response surface plot of the penetration and factors of influence.
(a) Contour plot between content of basalt fiber, content of diatomite and the penetration;
(b) Three-dimensional surface graph between content in basalt fiber, content in diatomite and the penetration.

In order to make a convenience for the analysis of the interaction of various factors on the penetration P, according to the test data, the three-dimensional response surface diagram and relevannt contour map (in the Fig. 1) of the penetration and independent variables are simulated. Keep the median value of one independent variable constant, and analyze the other two factors, thus, the optimal response value and the effective range of the respective variables are further determined. The two-dimensional contour plot is the projection of the three-dimensional response surface plot on the horizontal plane.

The apparent result can got from Fig. 1 (a) and (b) that: (1) when the content of basalt fiber is 3%, with the increasing content of diatomite, the change of bitumen penetration decreases sharply at first and then tends to be gentle. When the diatomite content increases from 0 to 12%, the degree of penetration decreases by 22.4%, diatomite increases from 11.8% to 15%, and the degree of penetration increases by 2.7%, it can be seen that adding diatomite will change the hardness and consistency of asphalt, and the greater the content of diatomite, the higher the hardness of asphalt. In the range of 0~12% diatomite content, with the advance of diatomite content, the penetration rate rises slowly from the minimum value. The analysis shows that diatomite is a light porous structure, and its strong adsorption can effectively combine with asphalt, reduce the light composition, form a relatively stable structure, increase the viscosity and slow down the fluidity of asphalt[19][20]. The addition of diatomite improves the asphalt's resistance to the high temperature deformation. (2) Within the same content of diatomite, the higher the content of basalt fiber, the lower the penetration. When the porportion of basalt fiber increases from 0 to 4% with the addition of 12% diatomite, the penetration decreases by 17.3%. Adding basalt fiber can obviously decrease the penetrating of asphalt mortar. Consequently, basalt fiber and asphalt contact surface constitute structural asphalt, and the formation of reinforced network structure can effectively share the stress with asphalt. (3) Both diatomaceous earth and basalt fiber can obviously reduce the penetration of asphalt mortar. The penetration of 11.8% diatomaceous earth and 3% basalt fiber modified asphalt mortar is 4.61mm, which is inferior to 5.2mm of SBS modified asphalt and 6.32mm of base asphalt.

The same test method can be obtained that the change of shear time has less influence on the penetration than diatomaceous earth and basalt fibers. In the case of large amount of diatomaceous earth and basalt fiber, within the increase of shear time, the penetration decreases slightly. For the sake of ensuring the uniform distribution of diatomaceous earth and basalt fiber in the asphalt, while considering shear efficiency, the proposed shear time is of 44min. Keeping a certain shearing time, the relationship between asphalt penetration and content of basalt fiber decreases linearly, within the increase of the content in diatomite, the penetration decreased obviously and then increased slightly. Due to the excessive amount of diatomite, the absorption of light components is saturated, and the excess diatomite particles will reduce the viscosity of asphalt.

### 3.4 Determination and Test Verification of Optimum Dosage

Through the above response surface test and variance analysis of fitting model, the optimal preparation scheme of modified asphalt can be obtained: when the content is 11.8% diatomite and 3.05% basalt fiber, and the shear rate is 4700r / min at 160 ℃ for 44.2min, the best three simulated indexes were the penetration of 4.62mm, the softening point of 59.1°C, and the ductility of 59.5mm at 5°C.Test verification was carried out under the condition of optimal dosageIn so as to proof that the simulation results of Design-Expert software is accurate. The measured results show that the penetration is 4.69mm, the softening point is 59.5°C, and the ductility is 59.2mm. Therefore, the model based on response surface method can better reflect the authenticity of the test.

## 4. Dynamic Shear Rheological Test

The test was carried out using dynamic temperature scanning, setting the test temperature and frequency from 58°C to 82°C and 10 rad/s. The rutting factor G*/sinδ was counted based on the results of the complicated modulus G* and phase angle δ. The test results were then compared to assess the viscoelastic change of the asphalt. Based on the consequences of the previous three major index and viscosity tests, base asphalt, 12% diatomite +3% basalt fiber modified asphalt, 15% diatomaceous earth +3% basalt fiber in modified bitumen and SBS modified bitumen were selected for dynamic shear rheology testing from 58°C to 82°C. The test consequences are as follows in Fig. 2.

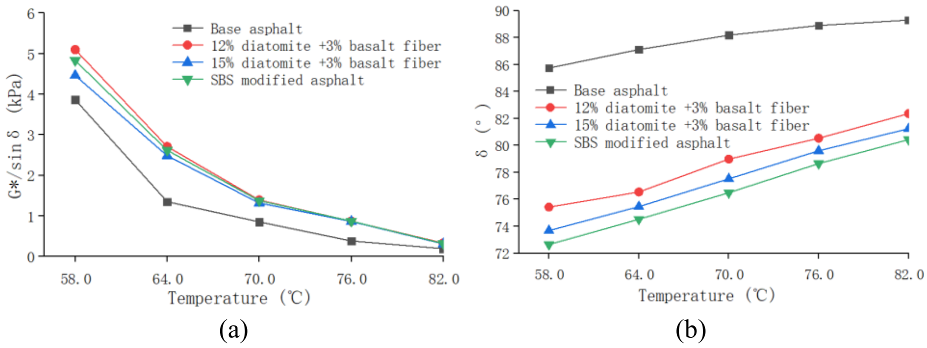



Fig. 2(a) Relationship between G*/sinδ and temperature, (b) relationship between δ and temperature

As seen in Fig. 2, the G*/sinδ of the bitumen and the temperature T follow the law of variation as a function G*/sinδ = Me-NT, where M and N are parameters. While the temperature is within the scope of 58°C to 76°C, G*/sinδ decreases substantially with increasing T. As asphalt is a material which is sensitive to the temperature, it becomes softer and less rigid as the temperature rises, and the inelastic component increases, making it more likely to deform. The phase angle δ is related to T by the variation δ = P×T+Q, where P and Q represent parameters, and the correlation between G*/sinδ, δ and T is shown in Table 8.

*Table 8*

**The correlation of G*/sinδ, δ and T**

| Type of asphalt | Rutting factor G*/sinδ=Me-NT | | | Phase angle δ=P×T+Q | | |
|---|---|---|---|---|---|---|
| Base asphalt | M=12419 | N=5.5637 | $R^2$=0.9954 | P=77.48 | Q=0.148 | $R^2$=0.9851 |
| 3% basalt fiber + 12% diatomite | M=2145 | N=9.6068 | $R^2$=0.9987 | P=57.91 | Q=0.297 | $R^2$=0.9969 |
| 3% basalt fiber + 15% diatomite | M=1071 | N=10.6214 | $R^2$=0.9978 | P=55.03 | Q=0.321 | $R^2$=0.9984 |
| SBS modified asphalt | M=1556 | N=10.0631 | $R^2$=0.9984 | P=53.55 | Q=0.328 | $R^2$=0.9992 |

The parameter N indicates the temperature sensitivity of the rutting factor. The parameter M reflects the degree of relationship between the rutting factor and temperature variation. As the data presented in Table 8: (1) The coefficient od association $R^2$ is greater than 0.98, which is a good correlation. (2) The relationship between M and N values is shown as follows: 3% basalt fiber +15% diatomite modified asphalt < SBS modified asphalt < 3% basalt fiber +12% diatomite modified asphalt <base asphalt.

The parameter P represents the sensitivity of the phase angle to temperature, the parameter Q represents the intercept with the phase angle coordinate axis. The P and Q values vary as follows: from SBS modified asphalt, 3% basalt fiber +15% diatomite modified asphalt, 3% basalt fiber +12% diatomite modified asphalt to base asphalt, the P and Q values increase sequentially. Apparently, the modified asphalt prepared together with 12% diatomite +3% basalt fiber has the best resistance in high temperature.

In order to research the aging behavior on both diatomaceous earth and basalt fiber compound modified asphalt, asphalt aging was simulated by rotary film oven test (RTFOT) and pressure aging test (PAV), rotary film oven aging test (RTFOT) can simulate the short-term aging behavior of asphalt in producing transportation and storage process.

The research presents that the rutting resistance factor of the compound modified asphalt after aging increases greatly, and the phase angle decreases. This is because after the asphalt is aged, the light elements in the asphalt components are continuously volatilized and polymerized into resins and further converted into asphaltenes, so that the viscous components in the asphalt decrease and the elastic components increase, what's more, it is able to advance the ability of modified asphalt in resisting deformation among high temperature. It shows that the incorporation of diatomaceous earth and basalt fibers could effectively enhance the anti-aging properties of asphalt.

## 5. Low Temperature Bending Rheological Test

Flow Testing (BBR) of curved beams proposed by SHRP Program. The dynamic rheological properties of various asphalts at low temperature were compared. The measurement results are as follows:

Referring from the Table 9: the lower the test temperature, the smaller the creep velocity of asphalt, the greater the stiffness modulus, when the temperature is set at -18℃, the stiffness modulus of base asphalt and 3% basalt fiber +15% diatomite is greater than 300MPa, it shows that asphalt is hugely influenced by temperature. Adding diatomaceous earth and basalt fiber could reduce stiffness modulus and increase creep rate. It shows that adding diatomite and basalt fiber has the ability to advance the low temperature deformation resistance of base asphalt in a certain degree, and the low temperature property of SBS modified asphalt is higher than that of basalt fiber and diatomite modified asphalt.

*Table 9*

**BBR Test Results of four types of asphalt**

| Type of asphalt | Stiffness modulus S(MPa) | | | Creep rate m | | |
|---|---|---|---|---|---|---|
| | -6℃ | -12℃ | -18℃ | -6℃ | -12℃ | -18℃ |
| Base asphalt | 67.2 | 189 | 360 | 0.446 | 0.286 | 0.182 |
| 3% basalt fiber + 12% diatomite | 32.5 | 141 | 282 | 0.525 | 0.352 | 0.294 |
| 3% basalt fiber + 15% diatomite | 34.2 | 154 | 304 | 0.501 | 0.329 | 0.279 |
| SBS modified asphalt | 20.8 | 89.3 | 232 | 0.493 | 0.387 | 0.275 |

## 6. Conclusion

For improving the comprehensive performance of diatomite-basalt fiber composite modified asphalt, a response surface design method was proposed, which combined quantitative analysis with qualitative analysis, and made up for the deficiency of traditional experimental design method. It meets the requirements of energy conservation, emission reduction and energy consumption reduction.

Taking the dosage of diatomite, basalt fiber and shear time as test factors, and the softening point, elongation at 5℃ and penetration degree as investigation indexes. The response surface design method is used for diatomite-basalt fiber composite modified asphalt formulation in multi-objective optimized designs, through analyzing the factors interactions, finally determine the optimum content of all the factors: when the content is 11.8% diatomite and 3.05% basalt fiber, and the shear rate is 4700r / min at 160 ℃ for 44.2min, compound modified asphalt owns the best resistence in high and low temperature.

Through DSR and BBR tests, comparing the base asphalt's resistence in high and low temperature, diatomite-basalt fiber composite modified asphalt and SBS modified asphalt, the study has showed that modified asphalt's resistence to high temperature with 12% diatomite and 3% basalt fiber is better than SBS modified asphalt, while its performance in low temperature is slightly worse than it.

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