Nano-particle's effect on silicone rubber inhibitor

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Abstract. There are several kinds of nanofiller's effect on silicone rubber inhibitors had been investigated. The result show that smaller diameter of particle will get better reinforcement effect, when 20nm Fe₂O₃ amount up to 10%, silicone rubber strength will get to 5.16Mpa. Different filler added into the silicone rubber show that quite different result, Fe₂O₃ is not only improve tensile strength but also enhance the heat stability of silicone rubber, and heat quality resume from 30.15% up to 86.15% at 500°C.

1. Introduction
Nano material is a kind of material which particles diameter between 1 ~ 100nm. Because of its average particle diameter is very small, so that has more surface atom, large specific surface, high surface energy and that make it quite different from single atom and its molecule different from common particle material. It has much property that common material dose not has, such as small size effect, surface effects. [1] Nano materials were widely used in rubber material modification recently. Comparison with traditional modification methods nanomaterial modification not only improve quality of rubber material but also gift rubber material many peculiar properties. It shows another way to polymer modification. So many researchers were proceeding in this field recent years.

The inhibitor plays an important role in propellant charge, it's main function to protect the motor from the heat and the erosion of hot-gas when propellant works [2~5]. Because of it's advantages of high decompose temperature, flame-resistance, antiablation and good compatibility with propellant charge, silicone rubber was widely used as inhibitor on propellant charge.

In order to make certain nanoparticle's effect on silicone rubber inhibitors property, several kinds of nanofillers were selected and added into silicone rubber inhibitors to find an answer.

2. Experimental

2.1. Raw materials
Silicone rubber(RTV): Chenguang chemical research institute; tetraethyl orthosilicate, dibutyltin dilaurate: Xian chemical reagent factory; nano-Fe₂O₃, nano-Al₂O₃: Nanjing Haitai nanomaterial; nano-TiO₂: Dalian luming; carbon black: Germany wacker; nanocarbon tube: Shenzhen Nano harbor Co, LTD.

2.2. Sample fabrication
Add weighed filler and addition agent in silicone rubber, dispersed 1h in rapid dispersion homogenizer, then add weighed curing agent tetraethyl orthosilicate and curing accelerator dibutyltin dilaurate, then
vacuum degassing 10 min before pouring into the mold, after 5 days curing in 35°C, then test the sample.

2.3. Sample test
Evaluation of mechanical property: Instron 6022 universal materials tester; executive standard: GB/T 528-1992; Sample preparation standard: GB528-76; Oxyacetylene ablation test instrument, 204 institute oxyacetylene ablation device; executive standard: GJB323A-96 SEM JEOL JSM-5800 executive standard: JY/T010-1996

3. Results and discussion

3.1. Comparison of dispersing craft
Comparison different of dispersing craft of nanofiller in silicone rubber inhibitor. The three-roll grinder was widely used in rubber industry, but when it was used in dispersion of nanofiller in silicone rubber we find that the result is worse than high-shear dispersion homogenizer. The high -shear dispersion homogenizer was made of stator and rotor, when rotor rotate during work, it absorbs silicone rubber into it's gap and because of high-shear actions make nanofiller was dispersed in silicone rubber inhibitor. The rotational speed of high-shear dispersion homogenizer can get up to 30000rpm. In the Table 1, the result of different dispersing craft impact on tensile strength was showed.

| Sample  | high-shear dispersion homogenizer | three-roll grinder |
|---------|----------------------------------|-------------------|
| A-1     | 0.91                             | 0.70              |
| A-2     | 1.14                             | 0.95              |
| A-3     | 1.82                             | 0.83              |
| Tensile strength (Mpa) | 387.6                             | 364.7             |
| Extensibility (%) | 297.4                             | 325.5             |

In the Table 1 A-1, A-2 and A-3 are content of nano-Fe$_2$O$_3$ from 5%, 10% to 15%. Dispersing time is 1h in high-shear dispersion homogenizer and 2h in three-roll grinder. From Table 1 we can see that the tensile strength of silicone rubber dispersed with high-shear dispersion homogenizer are better than three-roll grinder.

3.2. Nano and non-nanoparticle's reinforcement effect on silicone rubber:

|        | Fe$_2$O$_3$ | Al$_2$O$_3$ | TiO$_2$ |
|--------|-------------|-------------|---------|
|        | nano        | non-nano    | nano    | non-nano | nano    | non-nano |
| Tensile strength (Mpa) | 5.16       | 4.24        | 4.29    | 1.14     | 3.23    | 0.866   |
| Extensibility (%)   | 199        | 11.28       | 151.4   | 79.92    | 132     | 72.20   |

From Table 2 we can see that, non-nanoparticles’ reinforcement on silicone rubber inhibitor worse than nanoparticles’. It mainly because of non-nanoparticles’ bigger particles diameters and less surface inactivity, it makes silicone rubber molecule can’t effectively bonding with non-nanoparticles, so that less tensile strength was showed.
3.3. NanoFe₂O₃ particle's reinforcement effect on silicone rubber

From Figure 1, 20nm, 40nm Fe₂O₃ particle's effect on tensile strength of silicone rubber were studied. Figure 2 was NanoFe₂O₃ particles dispersion in silicone rubber (TEM). With 40nm Fe₂O₃ particles content increase we can see that the tensile strength of silicone rubber increases too. But with 20nm Fe₂O₃ particles content increase, the tensile strength of silicone rubber appears an extreme value at 10% quality content of 20nm Fe₂O₃ particles. Then with the content of 20nm Fe₂O₃ particles increased further, the tensile strength of silicone rubber decreased. It mainly because the high content of nanoparticle caused increase of viscosity, and that make nanoparticles uneven dispersing.

We think reinforcement mechanism of Nano Fe₂O₃ particles on silicone rubber mainly because its hydroxyl vibration peak in showed in 3400cm⁻¹ infrared diffuse reflectance (Figure 3), hydroxyl in nanoparticles and silicone rubber generate the interaction force play the role of reinforcement.

![Figure 1](image1.png)

**Figure 1.** NanoFe₂O₃ particles reinforcement effect on silicone rubber.

![Figure 2](image2.png)

**Figure 2.** NanoFe₂O₃ particles dispersion in silicone rubber (TEM).
3.4. Nano Fe$_2$O$_3$ particles effect on heat stability and antiablation of silicone rubber

Fe$_2$O$_3$ was widely used in silicone rubber as colorant and the amount is not large, but it will increase the heat stability of silicone rubber and form a harden carbon layer when silicone rubber was eroded by flame when rocket working.

**Figure 3.** Nano Fe$_2$O$_3$ particles infrared diffuse reflectance.

**Figure 4.** Nano Fe$_2$O$_3$ particles effect of heat stability on silicone rubber.
From Figure 4 we can see that nano-Fe$_2$O$_3$ particles dramatically improve the heat stability of silicone rubber inhibitor, when nano-Fe$_2$O$_3$ particles amount up to 10%, and resume quality from 30.15% up to 86.15% at 500°C.

Table 3. Nano Fe$_2$O$_3$ particles effect of antiablation on silicone rubber.

|                  | nanoAl$_2$O$_3$ | nanoFe$_2$O$_3$ | NMNT  | nanotube | Pure rubber | nanoTiO$_2$ |
|------------------|-----------------|-----------------|-------|----------|-------------|-------------|
| Ablation rate (mm/s) | 0.13            | 0.12            | 0.13  | 0.19     | 0.25        | 0.13        |

From Table 3 we know that when nanoparticles were added into silicone rubber, the antiablation rate all lower than pure silicone rubber. Among them, nano-Fe$_2$O$_3$ particles improve antiablation tare dramatically. It makes ablation rate of pure silicone rubber lower from 0.25mm/s to 0.12mm/s. From the sample test we find that during ablation test form a layer of carbon on silicone rubber surface and it keep the inner silicone rubber from the flame.

4. Conclusions
(1) From the test result we can clearly drew the conclusion that high-shear dispersion homogenizer in dispersion of nanofiller is better than three-roll grinder.
(2) Among all the selected nanoparticles the best reinforcement result was 20nm Fe2O3, when it's amount up to 10%, silicone rubber strength will get to 5.16Mpa.
(3) Nano-Fe2O3 particles dramatically improve the heat stability of silicone rubber inhibitor, when nano-Fe2O3 particles amount up to 10%, and resume quality from 30.15% up to 86.15% at 500°C.

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