Effect of nanosilica and nano zirconium oxide mixture modified by polydimethyl siloxane on the heat resistance of silicone paint

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ABSTRACT

Silicone-based paints are known for their high-temperature resistance and durability in chemical environments. This paper presents research findings on a silicone paint formulation supplemented with a mixture of nano-silica and nano-zirconium oxide, which has been surface-modified with Polydimethylsiloxane (PDMS). The incorporated ratio was 0.45% modified nano-silica and 1.05% modified nano-zirconium oxide. Following thermal endurance evaluation (a thermal shock test conducted at 1,050 °C for 25 seconds), no micro-cracks were observed on the surface. This is attributed to the increased flexibility of the silicone coating at high temperatures and the more uniform dispersion of the modified nano-additive particles within the silicone resin matrix, thereby enhancing the thermal resistance of the paint film. The author also conducted a comparative heat resistance test using an acetylene torch, with the sample plate's exposure temperature reaching approximately 1,100 °C for a duration of 25 to 30 seconds. The performance of the modified nano-particle-containing silicone paint was compared against the BKV inorganic heat-resistant paint and alkyd paint on steel test panels from a CT-18 engine, yielding favorable results.

Keywords: Silicone paint; Nanosilica; Nano zirconium oxide; Polydimethyl siloxane; High-temperature resistant paint.

1. INTRODUCTION

Silicone paint is a type of paint based on silicone resin. Silicone paint contains organic silane monomers, silicone, or silicone oligomers in the form of polymer coatings [1, 2]. The unique combination of silicone properties is well-suited for applications in the paint and coating field. Typically, paint can withstand temperatures of about 200 °C; however, if aluminum powder or silicates are added, it will create silicone paint with higher heat resistance, around 500 °C to 1000 °C [3-5]. During paint production, one or more types of additives are often added to increase thermal stability, improving the characteristics and applicability of the silicone paint film. Some authors have utilized nanosilica to enhance the thermal resistance of silicone resin-based paints, yielding promising results [6-10]. Other researchers have employed nano zirconium oxide to improve the adhesion, corrosion resistance, and thermal stability of paint films [11-15]. Besides, there are currently many methods to modify the surface of nanoparticles; however, after a specific analysis of each method, the author chose the method of modifying the nanoparticle surface with a silane coupling agent using PDMS. Then, nanosilica and nano zirconium oxide with modified surfaces will be used in different ratios to incorporate into the composition of heat-resistant silicone paint [16-18]. This study focuses on evaluating the effect of a modified nanosilica and nano zirconium oxide mixture on the heat resistance of silicone coatings.

2. EXPERIMENT

2.1. Materials

- Methylphenylsiloxane resin (trade name SILRES REN 50), origin Wacker Chemie AG (Germany);

- Aluminum paste (trade name STAPA 4 Aluminium Paste), origin ECKART GmbH (Germany);

- Nano zirconium oxide, origin American Elements (USA), fine powder, purity 99%, average particle size 60 nm;

- Nanosilica, origin Sigma-Aldrich (Germany), fine powder, purity 99.8%, average particle size 12 nm, specific surface area 175-225 m^2/g (by BET method);

- Polydimethyl siloxane (PDMS), origin Sigma-Aldrich (Germany), molecular weight: 2000 Da; viscosity: 20 cSt; density: 0.949 g/cm^3;

- Dimethyl carbonate (DMC), origin Aladdin (China), melting point 2-4 ^oC; boiling point 90 ^oC; density 1.069 g/cm^3; flash point 16 ^oC;

- Acetone, butanol, xylene, MIBK, bentonite: pure chemicals (China);

- TiO_2, origin Sichuan Lomon Corporation (China), particle size 10-15 μm; TiO_2 content ≥ 98%.

2.2. Paint preparation

Table 1. *Formulation for silicone paint preparation.*

No.	Components	Content (wt%)
1	Methylphenylsiloxane resin (SILRES REN 50)	40
2	Aluminum paste	15
3	Titanium dioxide	10
4	Modified Nanosilica	0 – 1.2
5	Modified Nano zirconium oxide	0 – 1.2
6	Bentonite	1
7	Acetone	5
8	Butanol	5
9	Xylene	10 – 7.6
10	Methyl isobutyl ketone	12
11	Additives	2

Prepare and quantify materials according to table 1. Soaking and aging stage: Add 90% Silres Ren 50 resin (polymethyl phenyl siloxane resin), 90% of MIBK solvent, and all remaining components into the stirring tank, stir evenly at a speed of 300-400 rpm for 1 hour. Soak and age the mixture for 24 hours. Grinding stage: Grind the mixture at a speed of 1300 – 1500 rpm, until the fineness is ≤ 25 μm. Adjustment stage: Add the remaining Silres Ren 50 resin and MIBK solvent, stir evenly at a speed of 300-400 rpm for 2 hours. Take paint samples for testing. Filtering - canning - storage stage: Use a 100 mesh/mm2 screen to remove all coarse particles or dirt in the paint, then proceed to canning and storage.

2.3. Analysis methods

After fabrication, silicone paint samples will be prepared (according to TCVN 2090:2007 standard) on steel plates according to TCVN 5670:2007 standard to evaluate the mechanical properties of coatings.

The heat resistance of the paint film was tested in a Nabertherm basic model N11/H furnace at the Measurement Center/Institute of Technology/General Department of Defence Industry.

The morphological structure of the paint material was observed on a High-Resolution Field Emission Scanning Electron Microscope (FESEM) Hitachi S4800 (Japan), magnification 2000 times, voltage 15 kV, at the Institute of Materials Science - Vietnam Academy of Science and Technology.

Thermal oxidation resistance analysis measurements were performed on a DTG-60H – SHIMADZU analyzer at the laboratory of the Faculty of Physical Chemistry – Hanoi National University of Education and a TGA - TG209F1 device, origin NETZSCH - Germany at the Institute of Materials Science/Vietnam Academy of Science and Technology.

The heat resistance of the paint film was tested using an acetylene torch according to ASTM-E285-08 standard, using a fixed oxy-acetylene torch, directly flaming the front and back surfaces of the coated sample plate. The maximum temperature of the oxy-acetylene torch flame can reach over 3000 ºC with an O_2/C_2H_2 ratio of 1/1.2. The jet flow rate is very high, perfectly suitable for testing the thermal protection and heat resistance of the coated sample plate. The coated sample plates were mounted on a temperature measurement test rig. A fixed oxy-acetylene torch was used, with the flame directly contacting the front surface of the coated sample panel. The distance from the torch tip to the sample panel was maintained at 10-20 cm. The temperature of test panels at the direct flame contact point and on the rear side of the panel, after a testing period of 20–25 seconds, was determined using a photometer (specifically, the handheld infrared thermometer OS524E-SC) (figure 1). Simultaneously, the changes in the sample panels before and after the test were evaluated to ascertain the thermal resistance of the paint film during the experiment.

Figure 1. *High temperature handheld infrared thermometer OS524E-SC.*

The schematic of the test method is presented in figure 2.

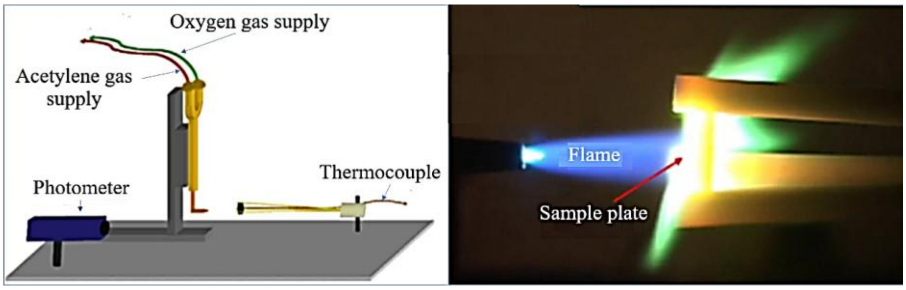


Figure 2. *Schematic of the heat resistance test of the coated sample plate using an oxy-acetylene torch according to ASTM-E285-08.*

Tested samples were evaluated for the degradation of the paint film after oven exposure and for the thermal resistance of the paint film under the influence of an acetylene torch flame. This evaluation was conducted according to TCVN 1205-2:2017.

3. RESULTS AND DISCUSSION

3.1. Effect of the content of modified nanosilica and nano zirconium oxide mixture on the heat resistance of the paint film

To evaluate the effect of the ratio of modified nanosilica and modified nano zirconium oxide on the heat resistance of the silicone paint film, silicone paint samples (with the main composition according to sample $M_{Al12Ti9}$) were prepared according to the formulas in table 2.

Paint samples corresponding to the above study formulations were prepared. Then, the test paint samples were prepared on steel plates (cut from CT-18 engine casings) following the sample preparation steps. The test samples were coated with a paint layer of 220 µm thickness, then dried to stabilize at room conditions for 24 hours before evaluating the heat resistance of the paint film at different temperatures in a Nabertherm 1300 ºC furnace for 25 seconds. The test results are shown in table 3.

Table 2. *Composition of silicone paint study formulations with different contents of modified nanosilica and nano zirconium oxide mixture.*

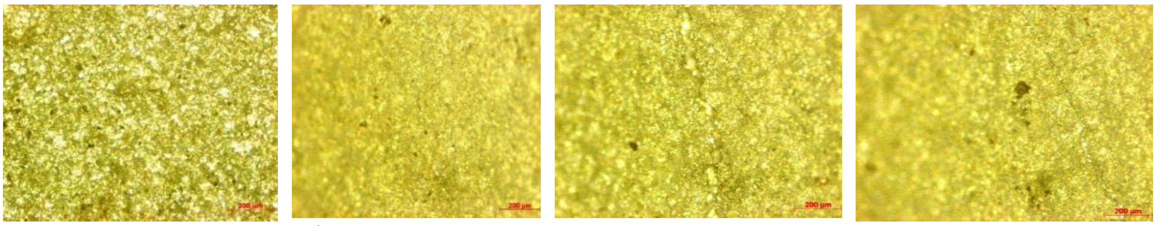
No.	Components	Contents (Wt. %)					
		$M_{Si0.75Zr0.75}$	$M_{Si0.75Zr0.75BT}$	$M_{Si0.25Zr1.25BT}$	$M_{Si0.45Zr1.05BT}$	$M_{Si1.05Zr0.45BT}$	$M_{Si1.25Zr0.25BT}$
1	Methylphenylsiloxane resin	56	56	56	56	56	56
2	Aluminum paste	12	12	12	12	12	12
3	TiO_2	9	9	9	9	9	9
4	Modified Nanosilica	-	0.75	0.25	0.45	1.05	1.25
5	Nanosilica	0.75	-	-	-	-	-
6	Modified Nano zirconium oxide	-	0.75	1.25	1.05	0.45	0.25
7	Nano zirconium oxide	0.75	-	-	-	-	-
8	Bentonite	0.5	0.5	0.5	0.5	0.5	0.5
9	Xylene	21.0	21.0	21.0	21.0	21.0	21.0
Total		100	100	100	100	100	100

Table 3. *Effect of the content of modified nanosilica and nano zirconium oxide mixture on the heat resistance of the silicone paint film.*

No.	Samples	Testing temperature (ºC)						
		600	700	800	900	1.000	1.050	1.100
1	$M_{Si0.75Zr0.75}$	UNC	UNC	UNC	UNC	UNC	UNC	BL
2	$M_{Si0.75Zr0.75BT}$	UNC	UNC	UNC	UNC	UNC	UNC	BL
3	$M_{Si0.25Zr1,25BT}$	UNC	UNC	UNC	UNC	UNC	BL	BL
4	$M_{Si0.45Zr1,05BT}$	UNC	UNC	UNC	UNC	UNC	UNC	BL
5	$M_{Si1,05Zr0.45BT}$	UNC	UNC	UNC	UNC	UNC	BL	PEE
6	$M_{Si1,25Zr0.25BT}$	UNC	UNC	UNC	UNC	UNC	BL	PEE

(UNC: unchanged; BL: Blasting; PEE: Peeling)

From the above test results, as the nanosilica/nano zirconium oxide ratio decreases, the heat resistance of the paint film increases; however, the heat resistance of the paint film begins to decline at a content ratio of 0.25/1.25. With nanosilica/nano zirconium oxide ratios of 0.75/0.75 and 0.45/1.05, the paint coating can withstand thermal shock at 1,050 ºC for 25 seconds. This can be explained by the fact that the nanosilica and nano zirconium oxide particles have been modified due to the chemical adsorption of PDMS oligomers carrying methoxy groups at the chain end onto the metal oxide surface. When these modified nanoparticles are added to silicone resin-based paint, the nanoparticles easily fill the voids in the silicone macromolecular chains due to their similar organic structure. This mechanism helps limit oxygen penetration and increases the heat resistance of the paint film. Besides, nano zirconium oxide powder has better heat resistance than nanosilica powder; therefore, paint films with a higher nano zirconium oxide ratio will have better heat resistance [8, 9, 19]. To evaluate the surface of the paint layer after the heat resistance test, the surfaces of the sample plates were observed under an optical microscope to assess the surface effect of the coating after the thermal shock process. Magnified images of the sample plate surfaces are shown in figure 3.



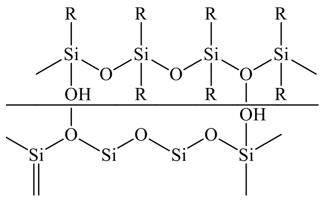
a. $M_{Si0.45Zr1.05BT}$ *– Before testing* *b.* $M_{Si0.45Zr1.05BT}$ *– After testing* *c.* $M_{Si0.75Zr0.75}$ *– Before testing* *d.* $M_{Si0.75Zr0.75BT}$ *– After testing*

Figure 3. *Optical microscope images of the paint film surface of sample plates before and after thermal shock testing*

The surface morphology and thermal resistance after thermal shock

Optical microscopy was used to examine the surface morphology of coated samples following thermal shock testing, assessing the impact of high heat on the coating surface. Results, also depicted in figure 3, illustrate heat-resistant paint samples with varying nano-particle ratios, which revealed clear distinctions in thermal endurance. Samples were evaluated included $M_{Si0.75Zr0.75}$: Silicone paint containing 0.75% unmodified nanosilica and 0.75% unmodified nano zirconium oxide. $M_{Si0.45Zr1.05BT}$: Silicone paint containing 0.45% modified nanosilica and 1.05% modified nano zirconium oxide. $M_{Si0.75Zr0.75BT}$: Silicone paint containing 0.75% modified nanosilica and 0.75% modified nano zirconium oxide.

Post-tested observations consistently indicated that both $M_{Si0.75Zr0.75}$ (unmodified) and $M_{Si0.75Zr0.75BT}$ (modified nanoparticles: 0.75/0.75) samples developed small cracks on their surfaces. In contrast, $M_{Si0.45Zr1.05BT}$, with its optimized ratio of 0.45% modified nanosilica and 1.05% modified nano zirconium oxide, showed no cracks on its surface. This demonstrates a clear hierarchy in thermal endurance: $M_{Si0.75Zr0.75}$ sample exhibited the lowest thermal endurance (most numerous cracks), followed by $M_{Si0.75Zr0.75BT}$ sample (some cracks). $M_{Si0.45Zr1.05BT}$ sample performed best, showing superior thermal shock resistance with an intact surface. This significant difference in surface structural morphology underscores the importance of both nanoparticle modification and optimal component ratio. Results establish 0.45% modified nanosilica and 1.05% modified nano zirconium oxide as the optimal ratio for enhancing the thermal resistance of silicone paint. Furthermore, the surfaces of paint samples became more uniform after the heat resistance test than before. This phenomenon can be attributed to the silicone paint layer becoming more flexible at high temperatures, allowing modified nano-additive particles to disperse more uniformly into the silicone resin matrix. Organic groups bonded to nanoparticle surfaces replace hydrophilic -OH groups present before modification, further contributing to the enhanced thermal resistance of the paint film [20]. The mechanism by which PDMS affects the dispersion and interaction with the silicone resin matrix, and consequently enhances the thermal shock resistance of the paint film, can be explained as follows: The surfaces of nanosilica and nano zirconium oxide particles contain hydroxyl groups, leading to an adsorbed moisture layer on their surfaces, approximately a few hundred Angstroms thick. This moisture layer is hardly removed even in a vacuum when heated to 400-500 °C. When nanosilica and nano zirconium oxide particles were incorporated into the paint film, an important phenomenon occurred at temperatures exceeding 400-500 °C. At these elevated temperatures, moisture adsorbed on the surface of nanoparticles is eliminated. Concurrently, hydroxyl groups (-OH) presented on nanoparticle surfaces can form bonds with free hydroxyl groups (those not yet bonded within macromolecular chains of silicone resin). This interaction led to an increase in the length of macromolecular chains. The extended polymer chains contributed to a more robust and interconnected network within the paint film, which in turn significantly enhances its thermal resistance.



For nanosilica and nano zirconium oxide particles that had been modified through chemical adsorption of PDMS oligomers (with methoxy end groups) onto their metal oxide surfaces, their incorporation into silicone resin-based paint films offers significant advantages. Due to their similar organic structure, these modified nanoparticles can readily fill voids within silicone macromolecular chains. This action effectively restricted the penetration of oxygen, which enhanced the thermal resistance of the paint film.

TGA Thermal Analysis

To evaluate the change in thermal properties of the paint film when using a mixture of modified and unmodified nanosilica and nano zirconium oxide powders, $M_{Si0.75Zr0.75}$ and $M_{Si0.45Zr1.05BT}$ paint samples were subjected to thermogravimetric analysis (TGA). The thermal analysis results are shown in figure 4.

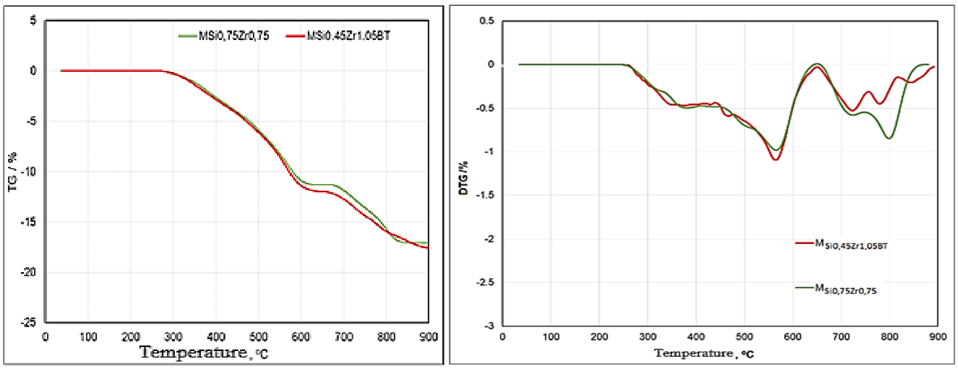


Figure 4. *Effect of the content of modified nanosilica and nano zirconium oxide mixture on the thermal properties of the paint film.*

The thermal analysis results indicate that the use of modified nanosilica and nano zirconium powder reduces the thermal properties of the paint film; however, the degree of reduction is very small. On the DTG graph, the peak representing the rate of mass loss decreases from 566.05 °C to 564.84 °C when replacing unmodified nano powder with modified nano powder. Besides, the ash content of the paint sample also decreased slightly, but not significantly. Specifically, the mass loss of the $M_{Si0.75Zr0.75}$ sample was 17.07%, while for the $M_{Si0.45Zr1.05BT}$ sample it was 17.46%. Thus, the thermal analysis results show that modifying the surface of nanoparticles when used in silicone-based paint can slightly reduce the thermal properties of the coating, but the level of change is insignificant [15, 20].

3.2. Heat resistance test of silicone paint film on steel sample plates

To evaluate the heat resistance of the heat-resistant silicone paint film before use for the CT-18 engine, the paint film was tested for heat resistance on steel sample plates under the impact of an acetylene torch flame for 25-30 seconds. Inorganic heat-resistant paint from Hanoi University of Science and Technology (BKV paint) and common alkyd paint on the market were also included in the study for comparison. The $M_{Si0.45Zr1.05BT}$ paint sample was coated on a steel sample (cut from the CT-18 engine casing) with a thickness of 200 µm, then dried according to the following procedure: Air dry naturally at room temperature for 10–12 hours; then raise the temperature from room temperature to different temperature levels: raise to 80 ºC and hold for 1 hour, raise the temperature to 120 ºC and hold for 1 hour, raise the temperature to 160 ºC and hold for 1 hour, finally raise the temperature to 200 ºC for 2 hours; then turn off the drying oven, let the sample cool slowly to room temperature. As for the BKV paint and alkyd paint samples, they were also coated on steel samples (cut from the CT-18 engine casing) with a thickness of 50-60 µm; however, the drying process was different to suit the properties of each product: air dry naturally for 10–12 hours at room temperature; then, raise the temperature from room temperature to 200 ºC with a heating rate of 2 to 3 ºC/minute, and maintain the temperature at 200 ºC for 2 hours; turn off the drying oven, let the sample cool slowly to room temperature. After the drying process, the outer paint layer of the steel samples was completely dry, with no blistering. The heat resistance of the paint layers was tested by 2 methods, presented in figure 5.

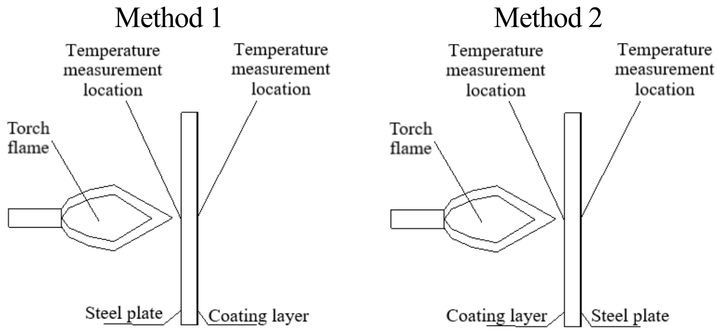


Figure 5. *Heat resistance test method using an acetylene torch.*

The results of evaluating the heat resistance of the paint samples were based on visual observation at positions around and in direct contact with the torch flame. The results are presented in table 4 and figure 6.

Table 4. *Results of heat resistance test of various paints under acetylene torch flame.*

No.	Sample name and test method	Temperature at flame contact point, ºC	Temperature behind sample plate, ºC	After testing
1	$M_{Si0.45Zr1,05BT}$ Method 1	1100	300-350	The paint film surface blistered and peeled off the substrate behind the flame contact point.
2	$M_{Si0.45Zr1,05BT}$ Method 2	1100	300-350	At the point of contact with the acetylene torch flame, the sample surface was carbonized, with large cracks appearing around it.
3	BKV coating Method 1	1100	300-350	The contact point behind the acetylene torch flame was discolored compared to the original, but no peeling occurred.
4	BKV coating Method 2	1100	300-350	The paint film surface at the point of contact with the torch flame was partially carbonized, without peeling or blistering.
5	Ankyd coating Method 1	1100	300-350	The paint film surface behind the flame contact point was completely carbonized and destroyed.

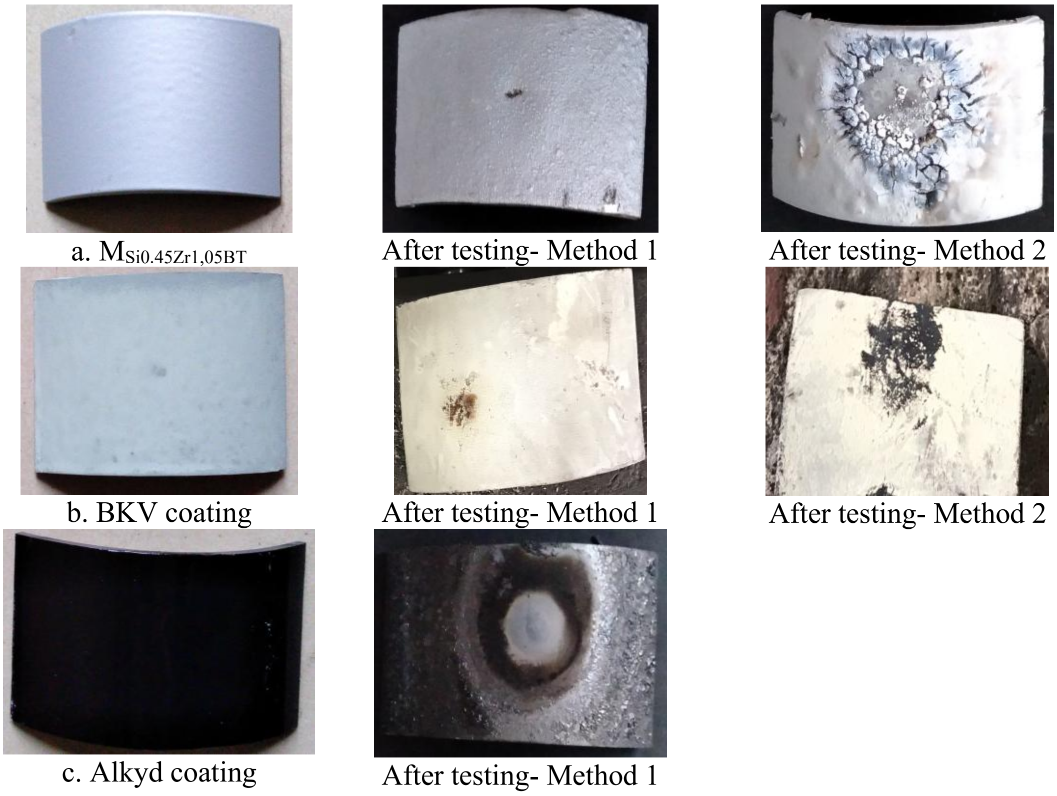


Figure 6. *Results of heat resistance test on sample plates using an acetylene torch flame for 3 different types of paint.*

The results from the test images on the sample plates of 3 different types of paint (heat-resistant silicone paint $M_{Si0.45Zr1.05BT}$, inorganic heat-resistant paint BKV, and alkyd paint) show that, for the heat-resistant silicone paint, at the point of direct contact with the acetylene torch flame (method 2), the paint film surface was almost completely destroyed. However, after testing according to method 2, the sample plates using heat-resistant silicone paint $M_{Si0.45Zr1.05BT}$ and inorganic heat-resistant paint BKV at positions around the direct flame showed that the paint layer blistered but was not destroyed. For the test according to method 1, the sample plate using common alkyd paint was destroyed and blistered; while the sample using heat-resistant silicone paint $M_{Si0.45Zr1.05BT}$ and inorganic heat-resistant paint BKV maintained a fairly good and comparable condition.

4. CONCLUSIONS

After the heat resistance test (thermal shock resistance test at 1,050 ^oC for 25 seconds), $M_{Si0.45Zr1.05BT}$ sample with a ratio of 0.45% modified nanosilica and 1.05% modified nano zirconium oxide did not show small cracks on the surface because at high temperatures, the silicone paint coating becomes more flexible and the modified nano additive particles disperse more uniformly into the silicone resin matrix, thereby increasing the heat resistance of the paint film. With its high thermal shock resistance and surface stability in high-temperature environments, this type of paint could be effectively applied to CT-18 engines. It offered excellent quality and had the potential to replace currently imported products.

Heat resistance using an acetylene torch was conducted (with a sample contact temperature of about 1100 ^oC) for (25-30) seconds for heat-resistant silicone paint $M_{Si0.45Zr1.05BT}$, inorganic heat-resistant paint BKV, and alkyd paint on CT-18 engine steel sample plates. Results indicated that: BKV paint had the best heat resistance, followed by the $M_{Si0.45Zr1.05BT}$ paint sample which had heat resistance nearly equivalent to BKV paint, and alkyd paint had the poorest heat resistance. It means that heat-resistant paints, especially BKV and $M_{Si0.45Zr1.05BT}$, have significant application potential for metal components exposed to high temperatures. This includes engine parts, industrial thermal equipment, and surfaces in direct contact with flame.

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TÓM TẮT

Ảnh hưởng của hỗn hợp nanosilica và nano ziriconi oxit biến tính bằng polydimetyl siloxan đến khả năng chịu nhiệt của sơn silicon

Sơn silicon được biết đến với khả năng chịu nhiệt độ cao và bền trong môi trường hóa chất. Bài báo này sẽ giới thiệu một số kết quả nghiên cứu về sơn silicon có bổ sung thêm hỗn hợp nanosilica và oxit nano ziriconi đã biến tính bằng Polydimetyl siloxan (PDMS với tỷ lệ 0,45% nanosilica biến tính và 1,05% nano zirconi oxit biến tính. Sau khi thử nghiệm khả năng chịu nhiệt (thử nghiệm khả năng chịu sốc nhiệt ở 1.050 ºC trong 25 giây) không xuất hiện các vết nứt nhỏ trên bề mặt do ở nhiệt độ cao lớp sơn phủ silicon trở nên linh hoạt hơn và các hạt phụ gia nano đã biến tính phân tán đồng đều hơn vào nền nhựa silicon, do đó làm tăng khả năng chịu nhiệt của màng sơn. Tác giả cũng tiến hành so sánh khả năng chịu nhiệt sử dụng đèn khò axetylen (với nhiệt độ tiếp xúc tấm mẫu khoảng 1100 ºC) trong thời gian (25 ÷ 30) của sơn silicon chịu nhiệt chứa các hạt nano đã biến tính với sơn vô cơ chịu nhiệt BKV, sơn ankyd trên tấm mẫu thép của động cơ CT-18 và thu được kết quả khả quan.

Từ khóa: Sơn silicon; Nanosilica; Nano ziriconi oxit; Polydimetyl siloxan; Sơn chịu nhiệt độ cao.