Research on the Effect of Molten Salt Ultrasonic Composite Cleaning for Paint Removal

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ABSTRACT: Aiming at paint removal on hydraulic cylinder, the effect of molten salt ultrasonic composite cleaning was studied. First, the mechanism of molten salt cleaning and ultrasonic cleaning was reviewed. To further describe the composite cleaning mechanism, the components and internal structure of paint were analyzed by scanning electron microscopy and Fourier transform infrared. Results showed that the paint had a significant layer structure. The total thickness was about 100 μm, and the main components were organic matters, including ester groups, epoxy groups, and aromatic compounds. Then, combining with thermal environment, cleaning medium’s property, and ultrasound, the composite cleaning mechanism was described in terms of three aspects: thermal effect, chemical reaction, and ultrasonic effect. Besides, the reason why this composite cleaning had good effect on paint removal, compared to paint heated in air, was explained through dynamic analysis, which was the reduction of reaction activation energy from 114.4 kJ/mol of paint alone to 74.1 kJ/mol.

1. INTRODUCTION

Under the effect of complex service environment and external field load, the surface of various mechanical products often forms pollution layers with strong adhesion and large coverage, such as carbon deposition inside the engine, carbonaceous pollutants inside the gearbox, and large areas of corrosion on part surface.¹ And to make mechanical structures possess anticorrosion and esthetic effects, surface coatings are usually added during the initial manufacturing stage, such as paint.² The surface coating of paint is widely used in many machines. When retired, some important parts of these machines are usually remanufactured so that the decommissioned products can be recycled and utilized to the maximum.³⁻⁵ However, the paint coated on the surface prevents the surface defects from being detected and repaired.⁶⁻⁷ Under the effect of air environment and external field load, some areas of the paint surface are broken and can easily generate electrochemical reaction, accelerating the corrosion of substrate. So, it is very vital to remove the paint coated on the surface to expose the substrate and the defects.

At present, the methods of paint removal are mainly thermal pyrolysis technology, laser cleaning, shot-blasting, organic solvent treatment, and dry ice treatment. The thermal pyrolysis technology generally uses high-temperature combustion to remove contaminants and can cause a lot of hot cracks on part surface; moreover, its energy consumption is very high and can generate many harmful substances that could cause serious pollution to the environment.⁸ Besides, while focusing the laser beam to vaporize the paint during the laser cleaning process, it could limit the purification efficiency of the harmful gas due to the small-size cleaning range of the laser beam, and the shot-blasting treatment generally uses small particles to shock the part surface to remove the paint, but the noise is very large during the cleaning process and could generate large amounts of dust.⁹,¹⁰ Organic solvent treatment utilizes the compatibility of the solvent with organic matters to peel off the paint from the part substrate, but the solvent itself and the falling paint fragments could cause immeasurable damage to the environment.¹¹ Dry ice treatment uses solid carbon dioxide to impart a certain kinetic energy to shock the paint surface at a high speed and makes the paint shrunk and embrittled by its instantaneous vaporization, but this treatment could exacerbate the greenhouse effect. However, molten salt cleaning has the characteristics of good fluidity and small surface tension and can react with paint at a high speed and absorb the harmful gases generated by chemical reaction.¹²,¹³ Besides, the introduction of ultrasound can increase the cleaning effect and accelerate the separation of the paint layer from the substrate.

At present, there has been much research on molten salt cleaning and ultrasonic cleaning. For molten cleaning, Forsberg, F used 80% sodium nitrate and 20% potassium nitrate (mass fraction) to carry out the cleaning experiments and introduced the current into the molten salt bath to remove the oxide layer.
on the alkali metal surface rapidly, where its voltage ranged from 6 to 12 V. The cleaning kinetics were explained as follows: the effect of the electric field increased the ion density on the part surface and made the ions arrange in order so that the reactions could be easily carried out.\textsuperscript{14,15} Yao used molten salt cleaning to remove the carbon deposition and explained the cleaning mechanism by combining with the physicochemical properties of molten salt, which included thermal expansion, surface tension, and chemical action.\textsuperscript{16} Nie found that the optimal cleaning parameters on carbon deposition removal were temperatures of 330–360 °C, NaOH mass fraction of 30%, and NaNO\textsubscript{2} mass fraction of 40% or more through carrying out the KNO\textsubscript{3}−NaNO\textsubscript{2}−NaOH ternary molten salt cleaning experiment.\textsuperscript{17} Guo investigated the viscosity and surface tension of the KNO\textsubscript{3}−NaNO\textsubscript{2} binary formula and found that the salt viscosity and surface tension decreased with the increase of temperature and the viscosity ranged from 2.3 to 3.8 cP at 190–250 °C.\textsuperscript{18} Long changed the cleaning temperature to sodium nitrate ratio when cleaning the dirt on the turbocharger center frame surface and established the regression equation and response surface model to quantitatively describe the cleaning cycle under different temperatures and sodium nitrate ratios. He also found that there were no adverse effects on cast iron and carbon steel under the cleaning parameters of $\omega$(NaNO\textsubscript{2})/ $\omega$(NaOH) = 1:1 (mass fraction) and cleaning temperature of 482 °C.\textsuperscript{19} For ultrasonic cleaning, Yusof N S M introduced physical and chemical effects in the ultrasonic process and believed that the acoustic cavitation process generated the acoustic streaming effect,\textsuperscript{20,21} and due to the action of the primary and secondary Bjerknes forces, the bubbles moved in the opposite direction of the pressure at a high speed, resulting in the development of bubble streamers. When the cavitation bubbles ruptured, high-intensity shock waves were generated and operated on the specimens’ surface.\textsuperscript{22} Meanwhile, the effect of acoustic cavitation led to the production of highly reactive radicals, which could act as a strong oxidant to oxidize the contaminants.\textsuperscript{23} Mazue used the 20 kHz ultrasonic system to clean contaminants in the bottom of the ship, made the cleaning process characterized, and optimized the parameters (distance, amplitude, and duration) of the hard matrix painting to determine the extreme cleaning efficiency.\textsuperscript{24} Maeno studied ultrasonic cleaning with a diluted hydrochloric acid solution to remove the thin oxide scale formed from the hot stamping of a bare 22 MnB\textsubscript{5} sheet. Through estimating the weldability and paint adhesion of surface-treated products, he found that the ultrasonic cleaning with diluted hydrochloric acid could successfully remove the thin oxide scale and found that the cleaned surface had sufficient quality for welding and painting.\textsuperscript{25}

In summary, many scholars had done a lot of research on the mechanism and applications of molten salt cleaning and ultrasonic cleaning and developed a complete technological system. However, the cleaning medium of ultrasonic cleaning was mainly water or solution. There is little relevant research on the molten salt medium with the introduction of ultrasound, and the effect of this composite cleaning method has not been studied.

In this paper, aiming at paint removal on hydraulic cylinder, the molten salt ultrasonic composite cleaning technology was studied. First, the mechanism of molten salt cleaning and the mechanism of ultrasonic cleaning were reviewed. To further describe the composite cleaning mechanism, the components and internal structure of paint were analyzed. Results showed that the paint had a significant layered structure. The total thickness was about 100 μm, and the main components were organic matters, including ester groups, epoxy groups, and aromatic compounds. Then, combining with the thermal environment, the cleaning medium, and ultrasound, the composite cleaning mechanism was described in terms of three aspects: thermal effect, chemical reaction, and ultrasonic effect. Through analyzing the relationship between the cleaning cycle, temperature, and ultrasonic power, it was found that that the effect of temperature on the cleaning cycle was higher than that of the ultrasonic power. But when paint was heated in air, which also had the thermal effect and reactions of pyrolysis and oxidation, the cleaning results were very poor. The reason was explained through the dynamic analysis, which was the reduction of reaction activation energy.

2. MECHANISM DESCRIPTIONS

Molten salt has had very broad applications for nearly a century, such as heat treatment, nuclear reactors, and so on. Owing to its low viscosity, low surface tension, and high specific heat capacity, molten salt was also introduced into the cleaning field to remove the contaminations on the part surface, such as paint and oxide scale.\textsuperscript{26} The mechanisms of molten salt cleaning were explained in terms of four aspects: thermal shock, wetting, deflocculation, and emulsification.\textsuperscript{27} When parts were put into a high-temperature cleaning environment, thermal shock produced and destroyed the adhesion strength of contaminants on the substrate. The wetting effect could make the salts delivered into the contaminants as well as the interface between contaminants and substrate. The deflocculation effect could break down the solid or semi-solid contaminants into small particles. And the emulsification effect could cause the detachment of oily films from substrates.\textsuperscript{18} Besides, when contaminants were mainly organic coatings, oxide scales, and carbonized residue, some chemical reactions were also present in the cleaning system, as shown below.\textsuperscript{28,29}

\[
\begin{align*}
(C_{n}H_{2}O_{n})_{m} + \frac{4x + y}{2}O_{2} &\xrightarrow{800F}\ nxCO_{2} + \frac{ny}{2}H_{2}O \\
(C_{n}H_{2}O_{n})_{m} &\xrightarrow{800F}\ nC_{n}H_{2-z/2} + nzH_{2}O \\
CO_{2} + 2OH^{-} &\xrightarrow{800F}\ CO_{3}^{2-} + H_{2}O \\
R_1 - COO - R_2 + OH^{-} &\xrightarrow{800F}\ R_1 - COO^{-} + R_2 - OH \\
R_2 - OH + O_{2} &\xrightarrow{800F}\ CO_{2} + H_{2}O \\
C + 2NaNO_{3} &\xrightarrow{800F}\ CO_{2} + 2NaNO_{2} \\
2AO + 2MO + 2NaNO_{3} &\xrightarrow{800F}\ A_2O_3 + M_2O_3 + 2NaNO_2 \\
2M_2O_3 + 4NaOH &\xrightarrow{800F}\ 2Na_2M_2O_7 + 2H_{2}O
\end{align*}
\]

Ultrasonic cleaning is used widely in all walks of life. It uses the ultrasonic waves generated by electromagnetic oscillator to operate on the part surface and achieve the purpose of removal.\textsuperscript{30} The high-frequency oscillations generated by the ultrasonic wave made the cleaning medium generate cavity bubbles of near vacuum. When the sound pressure or sound intensity reaches a certain value, the bubbles rapidly grow and suddenly close with shock waves generated instantly. Then, a pressure of about 1012 Pa was generated,\textsuperscript{31} which destroyed the structure and the adsorption of insoluble dirt. The continuous
cavity bubbles, breaking the contaminants' surface, causing fatigue damage of the dirt layer. For oily contaminants, the effect of emulsification occurred by high-frequency oscillation and high pressure, dispersing the oil into the medium. So, the solid particles adhered to the specimens’ surface by oil stain peeled off. When the intrinsic frequency of cavity bubbles equals the vibration frequency of ultrasonic wave, the resonance effect can be induced, leading to the accumulation of large amounts of heat energy around the cavity bubbles, breaking the contaminants' chemical bond and eventually resulting in the separation between contaminants and substrate. Overall, molten salt cleaning and ultrasonic cleaning had good removal effects on contaminants. When ultrasound was introduced into molten salt cleaning, both cleaning effects would be enhanced and this composite cleaning contained, above all, removal effects of two methods. To further describe the composite mechanism on paint removal, a composite cleaning experiment was carried out in the cleaning environment.

3. EXPERIMENTAL SECTION

3.1. Materials. 3.1.1. Formula. By analyzing the literature on the molten salt cleaning and ultrasonic cleaning, it can be found that the key process parameters for paint removal are cleaning temperature and ultrasonic power. According to the previous research on the optimization of salt formula, melting temperature, cleaning effect, and viscosity, the salt formula in this system is m(NaNO₃)/m(KNO₃)/m(NaOH)/m(NaNO₂) = 35:28:30:7%. All of the salts in the formula are analytically pure and were produced by Sino-pharm Group Chemical Reagent Co. Ltd.

3.1.2. Hydraulic Cylinder. The hydraulic cylinder is one of the most representative mechanical parts with paint coating. The paint on the part surface is thick, uniform, and easy to be divided into equal areas. The adhesion strength is very high, preventing from oxidation damage. So, the hydraulic cylinder is taken as the cleaning objective. The hydraulic cylinder from the retired XCMG Excavator XE60D and the material is mainly 1145 carbon steel. For the convenience of the follow-up cleaning experiment and the scanning electron microscopy (SEM)/Fourier transform infrared (FTIR) analysis, the whole hydraulic cylinder is equally divided into eight parts on one circumference using the Wire Cut Electrical Discharge Machining (DK773S, made by Hua Zheng), and the width of the part is 120 mm. The whole hydraulic cylinder and one of the cut specimens are shown in Figure 1, which are shot by Canon EOS 80 D. The parameters of lens are EF-S of 18–135 mm and f/3.5–5.6.

3.2. Test Equipment. The test equipment uses a self-made molten salt ultrasonic composite cleaning machine. The machine is equipped with double cleaning tanks, including the molten salt ultrasonic composite cleaning tank and an ultrasonic water-washing tank, as shown in Figure 2. The electronic cabinet controls the ultrasonic power and cleaning temperature. When temperature reaches the preset parameters, the electronic control cabinet is intelligently turned on and off so that the temperature in the tank is controlled within a preset range. Furthermore, the ultrasonic frequency is fixed at 28 kHz, the number is 24, the power adjustment range is 0–1440 W, and the cleaning temperature range is 0–500 °C with precision ±1 °C. The ultrasonic oscillators are symmetrically arranged on the front and back surfaces of the cleaning tank.

3.3. Analysis of the Component and the Internal Structure of the Paint. 3.3.1. Energy Dispersive X-ray Spectroscopy (EDS) Combination Analysis. The internal structure of paint was investigated using the scanning electron microscope modules at an accelerating voltage of 10 kV. Then, each layer was scanned by energy-dispersive spectrometer modules to obtain the elements of each paint layer. Before SEM, all samples were mixed with the mosaic powder (urea—formaldehyde molding powder, made by SHSIIW) and then made into a mosaic block, which could be conductive.

3.3.2. Infrared Spectra Analysis. The paint debris obtained from the specimen’s surface was ground into powders. Then, the paint powders (ca. 5 mg) were mixed with 150 mg of KBr and milled thoroughly to reach a particle diameter of < 2.5 mm. The mixtures were compressed into pellets under approximately 10–12 MPa and analyzed using a VERTEX-70FTIR spectrometer. Spectra were recorded at a resolution of 2 cm⁻¹ for 400–4000 cm⁻¹.

3.4. Analysis of the Viscosity of Molten Salt. The mixed salt was put in the testing room. The temperature was raised to 350 °C, and the viscosity was measured after holding for 25 min. Then, the temperature reduction program was performed and the system temperature was lowered to 340 °C. After temperature holding for about 25 min, the viscosity value was measured. The above procedures were repeated until the temperature declined to 190 °C.

3.5. Analysis of the Components of the Gas. The analysis equipment was a gas chromatograph—mass spectrometer (GC–MS) (7890B-5977A, made by Agilent). And the test condition was shown as below.

3.5.1. Chromatographic Conditions. The column was a PLOT-Q capillary column; the inlet temperature was 250 °C; the initial temperature was 35 °C; the heating rate was 10 °C/min; the termination temperature was 200 °C; the retention time was 20 min; the carrier gas was high-purity helium (99.9995%); and the flow rate was 0.8 mL/min.

3.5.2. Mass Spectrometry Conditions. Ionization mode was ion source; ionization energy was 70 eV; the ion source temperature was 230 °C; the quadrupole temperature was 150 °C; the transmission line temperature was 290 °C; the multiplication tube voltage was 1.623 V; and the scanning mass range was 50–700 amu.

3.6. Thermogravimetric (TG) Analysis of Paint, Salt, and Their Mixture. The thermogravimetric analysis equipment was Swiss METTLER TOLEDO TGA1 thermogravimetric analyzer. The carrier gas was air. The temperature range was 25–800 °C, and the heating rate was 10 °C/min. The paint was ground to a powder. All specimens’ composition and mass, which were analyzed by a thermogravimetric experiment, are shown in Table 1.

Figure 1. Cleaning objective.
3.7. Cleaning Procedures. The composite cleaning test adopted the central composite test method. The independent variables are cleaning temperature and ultrasonic power. The dependent variable is the cleaning cycle. According to test equipment parameters and the literature, it was determined that the cleaning temperature $T$ range was $270 - 340 \, ^{\circ}\mathrm{C}$ and the ultrasonic power $P$ range was $1000 - 1440 \, \text{W}$, and the test factors and levels are shown in Table 2. The cleaning procedure is summarized in Figure 3.

- **(1)** This experiment used the central composite design to carry out this research. According to the 13 sets of test parameters set by Minitab, the hydraulic cylinder was divided into 13 parts and the specimens were numbered separately.
- **(2)** First, the salt was placed in the composite cleaning tank, which was configured according to the formula, and it was fully melted. Meanwhile, the cleaning parameters were set according to the requirements. After putting the specimens into the cleaning tank, the time was recorded rapidly. The experimental phenomenon could be observed that bubbles occurred during the cleaning process and the fragments fell off from the specimens. When the reaction phenomenon stopped, the cleaning was over. Then, the final time was recorded immediately.

### Table 1. Specimens’ Number, Composition, and Mass

| specimens | composition                        | mass (mg) |
|-----------|------------------------------------|-----------|
| 1         | paint                              | 2         |
| 2         | molten salt                        | 10        |
| 3         | mixture of molten salt and paint   | 8 + 2     |

### Table 2. Test Factors and Levels

| element          | low  | high |
|------------------|------|------|
| temperature $T$ ($^{\circ}\mathrm{C}$) | 270  | 340  |
| ultrasonic power $P$ (W)           | 1000 | 1440 |

4. ANALYSIS AND RESULTS

4.1. Physicochemical Properties of Paint. 4.1.1. Micro-morphology Analysis. The paint surface and profile morphology are shown in Figures 4 and 5, respectively. The surface morphology showed that the paint surface was flat at 500 times, with even dark stripes and dense white spots. At 10 000 times, it can be further seen that the white spots were densely arranged on the surface and varied in size and there were no obvious pits and cracks. On further observation at 20 000 times, results showed that the white particles ranged from 200 to 600 nm and were irregular. According to the SEM imaging principle, the shiny white particles were doped metal particles in the paint.

The profile morphology showed a distinct layered structure in the paint at 500 times magnification. The total thickness of the paint layer was about $100 \, \mu\text{m}$, as shown in Figure 5a. When further enlarged to 1000 times, as shown in Figure 5b, it showed obvious structural differences. The topcoat was compact and flat. The primers were mostly long strips of particles that were significantly larger than the particles in the topcoat and tightly adhered to the substrate.

4.1.2. Elements Analysis. To analyze the components of each layer, EDS was used. The elements and mass fraction of the topcoat and the primer are shown in Tables 3 and 4, respectively. Results showed that there were many kinds of elements in the paint, but the content was not uniform. Both in the topcoat and primer, the proportion of C and O elements was the highest,
over 70%, which indicated that the main component of paint was organic matter. In addition to C and O elements, the paint also contained metal elements such as Al, Ti, Cr, Fe, etc. This was mainly due to additives such as anticorrosive ones. The Si may be due to the doping of dust particles in the paint. Pb is the harmful heavy-metal element derived from the colorant in the paint, which was the third highest element in paint.

4.1.3. FTIR Analysis. From the elements analysis, it could be seen that the main components of the paint were organic matters. To further study the internal groups of the paint, the
primer and topcoat were measured by FTIR spectroscopy, and the results are shown in Figure 6. According to the FTIR spectrum of the topcoat in Figure 6a, the peaks at 1725 and 1687 cm⁻¹ indicated that the topcoat contained an ester group (−COO−), which could use the alkaline cleaning environment to promote the breakage of the ester group and then be decomposed. The 1074, 763, and 700 cm⁻¹ peaks indicated that the topcoat contained aromatic compounds.

Figure 6b of the primer spectrum showed that the absorption peak appeared near the primers at 912 and 828 cm⁻¹, indicating that the primer contained the epoxy group. The absorption peaks at 1447 and 1508 cm⁻¹ indicated that the primer may contain benzene rings, while the peaks at 1040 and 1017 cm⁻¹ were characteristic peaks of aromatic ethers and esters, thus demonstrating that the primer contained a benzene ring structure.

According to the above analysis, the main components of the paint were organic matter, containing ester groups, epoxy groups, and aromatic compounds, which showed a distinct layered structure in the paint. The topcoat structure was firm and flat. The primer particles were large and strongly adhered to the substrate surface. Due to the presence of harmful heavy-metal elements in paint, attention should be paid to the treatment of these elements.

4.2. Mechanism of the Composite Cleaning for Paint Removal. To describe the mechanism of this composite cleaning, the viscosity of molten salt and the gaseous production were analyzed. Combined with the components of paint and the mechanism of molten salt cleaning and ultrasonic cleaning, the removal mechanism was focused on the effects of thermal environment, chemical reaction, and ultrasound.

4.2.1. Thermal Effect. In the cleaning system, all of the contaminants and substrate were dipped into the cleaning medium and were all under the effect of high temperature. However, the thermal expansion coefficients of the metal substrate and paint were different. At 20−400 °C, the expansion coefficient of steel was (12.9−13.9) × 10⁻⁶. Because of the organic components, the paint expansion coefficient was much larger than that of steel. The difference between the two materials’ expansion coefficient makes a certain degree of extension on the steel substrate, resulting in some cracks in the interior of contaminations. Meanwhile, due to the mainly organic components of the paint, it can be softened under the thermal effect, and a certain amount of gullies are generated on the paint surface so that the iron can easily adhere to the ion-containment reaction interface and participate in the reaction. The change of paint on the substrate surface is shown in Figure 7. Furthermore, the thermal effect can provide the activation energy required for the hydrolysis reaction of OH⁻ with ester groups and the oxidation reaction.

On the other hand, the thermal effect on the cleaning system can effectively make the cleaning medium obtain a very low viscosity. Figure 8 shows that within the range of 350 °C, the viscosity would decline with the increase of temperature. When the system temperature ranged from 200 to 350 °C, the viscosity of molten salt kept within 3 cP, indicating that the cleaning medium kept high fluidity. High fluidity could make it easier to remove the contaminants from the surface and prevent the contaminants’ fragment from attaching to the substrate again. Moreover, high fluidity could also enhance the ion migration rate, replenishing the loss of ions on the reaction interface and making the ions uniform in the cleaning system. Because of the salt’s physical properties of small surface tension in the thermal environment, it could allow the medium to obtain low surface tension in this composite cleaning system. With low surface tension, it was easy to combine the cleaning medium with the paint surface and infiltrate interior to the paint, making the chemical reaction easy on the medium-containments interface. Under the effect of low surface tension and viscosity, the ion-exchange rate on the reaction interface would be accelerated so that the NO₃⁻, O₂⁻, and OH⁻ can react with the ester group, epoxy groups, and the aromatic compounds at a high speed. The cleaning medium could also permeate into the interior of the paint through the cracks and react with the internal contaminants, leading to escape of gaseous production from the internal paint and producing expansion forces to intensify the thermal expansion.

4.2.2. Chemical Reaction. When conducting the cleaning experiment for paint removal, there were many bubbles around the paint that escaped from the cleaning system. Some fragments of paint were peeled off and gradually decomposed. So, there must be some chemical reactions existing in the cleaning system. Due to the thermal environment, the cleaning medium was full of ions, such as NO₃⁻, OH⁻, CO₃²⁻, and so on. The presence of NO₃⁻ and CO₃²⁻ enhanced the solubility of oxygen, which existed in the form of O₂²⁻ and O₂⁻ in the cleaning system. These ions could chemically react with the organic components. Moreover, the thermal environment provided the activation energy of the chemical reaction. The removal model of chemical effect is shown in Figure 9.

To investigate the reactive versions, the gaseous production was analyzed by gas chromatography–mass spectrometry. The approximate composition and content of gaseous production
The reason was that due to the presence of NO₃⁻ containments in the cleaning system were completely oxidized during the cleaning process and proved that some of the O₂ could undergo a violent oxidation reaction under the influence of the ultrasonic oscillators located on both sides of the cleaning tank can continuously generate ultrasonic waves and high-frequency oscillations during its operation. On the one hand, the fluidity of the medium can be enhanced, resulting in the uniform distribution of the internal temperature field and ions concentration.

The ultrasonic oscillators located on both sides of the cleaning tank can continuously generate ultrasonic waves and high-frequency oscillations during its operation. On the one hand, the fluidity of the medium can be enhanced, resulting in the uniform distribution of the internal temperature field and ions concentration. Owing to the uniform ion concentration and the enhancing fluidity, the ion-exchange rate at the medium-containments reaction interface is accelerated so that the ions at the reaction interface can be quickly replenished and then the reaction can be maintained at a high speed. Meanwhile, the oscillation effect can also make the contaminant fragments move around, preventing them from falling onto the paint surface to reduce the reaction rate. It could be obviously seen that there were some ripples on the interface of the cleaning system and the fragments of paint were moved around, which indicated the enhanced fluidity of the cleaning medium.

On the other hand, the high-frequency oscillations make the cleaning medium generate cavity bubbles of near vacuum. When the sound pressure or sound intensity reaches a certain value, the bubbles grow rapidly and then suddenly close. Therefore, the shock wave is generated at the moment, acting on the paint surface and causing more cracks. The adhesion of the paint is destroyed. Moreover, the continuous closing and breaking of the

were obtained as shown in Table 5. Results showed that including CH₄, the total amount of complex organic gases and CO was only 4.81%, indicating that it would pay a little cost to eliminate the negative influence on the environment caused by this composite cleaning technology (Figure 10).

As for the complex gas, its components and contents were obtained through comparing the GC–MS analysis results with the standard library NIST 14, as shown in Table 6. Results showed that there were 18 kinds of compounds in complex gas, including seven kinds of hydrocarbons, four kinds of hydroxyl, and ester were mainly derived from the pyrolysis of paint. The aldehydes and ketones were mainly derived from incomplete oxidation of the hydroxyl. And a large amount of nitrate ions could be used as a reaction catalyst to continuously provide superoxide ions to oxidize the containments. So, the organic components in the paint, such as the ester groups, epoxy groups, and the aromatic compounds, could undergo a violent oxidation reaction under the effect of O₂. With the synergistic effect of pyrolysis and oxidation, the paint was further decomposed, making the interior of the paint loosened and reducing the paint adhesion strength. So, the reactions of pyrolysis and oxidation were involved in paint removal. The reaction equations are shown below

\[
\begin{align*}
\text{C}_{(2x+2)}\text{H}_x\text{O}_{2z} & \xrightarrow{300^\circ C} 2\text{C}_x\text{H}_y + z\text{CO}_2 \\
\text{R}_1 - \text{COO} - \text{R}_2 + \text{OH}^- & \xrightarrow{300^\circ C} \text{R}_1 - \text{COO}^- + \text{R}_2 - \text{OH} \\
\text{CO}_2 + 2\text{OH}^- & \xrightarrow{300^\circ C} \text{CO}_3^{2-} + \text{H}_2\text{O} \\
\text{O}_2 + 2\text{CO}_3^{2-} & \xrightarrow{300^\circ C} 2\text{O}_2^{2-} + 2\text{CO}_2 \\
3\text{O}_2 + 2\text{CO}_3^{2-} & \xrightarrow{300^\circ C} 4\text{O}_2 + 2\text{CO}_2 \\
\text{O}_2^{2-} + 2\text{NO}_3^- & \xrightarrow{300^\circ C} 2\text{O}_2 + 2\text{NO}_2 \\
\text{NO}_2^- + 2\text{O}_2 & \xrightarrow{300^\circ C} \text{NO}_3^- + \text{O}_2 \\
4\text{C}_x\text{H}_y + (4x + y)\text{O}_2 & \xrightarrow{300^\circ C} 4x\text{CO}_2 + 2y\text{H}_2\text{O} \\
(\text{C}_x\text{H}_y\text{O}_z)_a & + n\left(2x + \frac{y}{2} - z\right)\text{O}_2 & \xrightarrow{300^\circ C} nx\text{CO}_2 + \frac{ny}{2}\text{H}_2\text{O} \\
\text{R} - \text{CH}_2 - \text{OH} + \frac{1}{2}\text{O}_2 & \xrightarrow{300^\circ C} \text{R} - \text{CHO} + \text{H}_2\text{O} \\
\text{R}_1\text{R}_2 - \text{CH} - \text{OH} + \frac{1}{2}\text{O}_2 & \xrightarrow{300^\circ C} \text{R}_1 - \text{CO} - \text{R}_2 + \text{H}_2\text{O}
\end{align*}
\]

In addition, these complex gases were just 4.73% of the total gas. It could be easily treated by exhaust gas treatment appliance. Results of Tables 6 and 7 showed that the gaseous components did not contain any metal elements. They still remained in the molten salt system. After cleaning, the cleaning medium gradually solidified into blocks with the decrease of temperature. So, these heavy-metal elements could be fixed in the salt blocks and be easily treated in the post-treatment process.

4.2.3. Ultrasonic Effect. The ultrasonic oscillators located on both sides of the cleaning tank can continuously generate ultrasonic waves and high-frequency oscillations during its operation. On the one hand, the fluidity of the medium can be enhanced, resulting in the uniform distribution of the internal temperature field and ions concentration. Owing to the uniform ion concentration and the enhancing fluidity, the ion-exchange rate at the medium-containments reaction interface is accelerated so that the ions at the reaction interface can be quickly replenished and then the reaction can be maintained at a high speed. Meanwhile, the oscillation effect can also make the contaminant fragments move around, preventing them from falling onto the paint surface to reduce the reaction rate. It could be obviously seen that there were some ripples on the interface of the cleaning system and the fragments of paint were moved around, which indicated the enhanced fluidity of the cleaning medium.

| components | H₂ | O₂ | N₂ | CO | CH₄ | CO₂ | complex gas |
|------------|----|----|----|----|-----|-----|-------------|
| mass fraction (%) | 0.96 | 19.01 | 74.68 | 0.08 | 0.04 | 0.50 | 4.73 |

Figure 9. Removal model of the chemical effect.
cavity bubbles repeatedly shocked the paint surface, causing fatigue damage of the paint and further extension cracks.42

Combined with components of the paint and the mechanism of molten salt cleaning and ultrasonic cleaning, the composite cleaning mechanism could be divided into three aspects: ultrasonic effect, thermal effect, and chemical reaction. The high-temperature environment in the system caused the paint to undergo a certain degree of thermal pyrolysis and provided the activation energy that was required for the oxidation reactions. Besides, with the introduction of ultrasound, it could enhance the fluidity of the cleaning medium. Owing to the high-frequency oscillation, the ion concentration and temperature were uniform in the system and the high-frequency oscillations led to the generation of ultrasonic cavitation effects that shocked the paint and therefore destroyed its adsorption.

4.3. Dynamics Analysis. When carrying out composite cleaning, the cleaning cycle $t$ was recorded under each cleaning parameter of temperature and ultrasonic power. Results are shown in Table 7.

Table 7 shows that with the increase of temperature and power, the cleaning cycle was gradually shortened. Temperature was the precondition of the thermal effect and provided the activation energy required by chemical reactions. Ultrasonic power directly affected the ultrasonic removal effect. To further illustrate the relationship between cycle, temperature, and power, Minitab 17.0 was used to generate the respond surface figure, as shown in Figure 11. Results showed that when power was fixed, the cycle would change significantly along with the increase of temperature. But when temperature was fixed, there was a small change of cycle with the increase of power. So, the effect of temperature on the cleaning cycle was larger than that of ultrasonic power. Through the response variable optimizer in Minitab, the optimal temperature was obtained as 335 °C and power was 1440 W (the maximum of the equipment). The optimal cycle was 4.5061 min.

Table 6. GC−MS Qualitative and Area Ratio of Gas Product

| components                               | mass fraction (%) |
|------------------------------------------|-------------------|
| (hexanal)C$_6$H$_{12}$O                  | 1.10              |
| (isovaleraldehyde)C$_7$H$_{10}$O         | 0.66              |
| (acetone)C$_3$H$_6$O                     | 2.28              |
| (2-octylene)C$_{10}$H$_{16}$             | 0.74              |
| (hexamethylcycloctoxane)C$_{16}$H$_{18}$O$_3$Si$_3$ | 2.19          |
| (butyraldehyde)C$_3$H$_6$O               | 0.97              |
| (ethyl acetate)C$_7$H$_{10}$O            | 14.22             |
| (2-butane)C$_3$H$_6$O                    | 0.41              |
| (benzene)C$_6$H$_6$                      | 11.84             |
| (2-ethyl propylene aldehyde)C$_3$H$_6$O  | 4.28              |
| (methyl methacrylate)C$_3$H$_5$O         | 19.02             |
| (methylbenzene)C$_6$H$_4$                | 3.89              |
| (2,3,4-trimethyloxetacyclic butane)C$_8$H$_8$O$_2$ | 26.47          |
| (butyl acrylate)C$_3$H$_6$O$_2$O          | 1.41              |
| (decamethylcyclopentasiloxane)C$_{10}$H$_{30}$O$_3$Si$_5$ | 0.79          |
| (styrene)C$_3$H$_6$                      | 8.54              |
| (cis-2-pentene-1-alcohol)C$_{10}$H$_{18}$O | 0.80          |
| (benzaldehyde)C$_3$H$_4$O                 | 0.40              |

Table 7. Cleaning Cycle Under Different Cleaning Parameters

| number | temperature (°C) | ultrasonic power (W) | cleaning cycle $t$ (min) |
|--------|------------------|----------------------|-------------------------|
| 1      | 329.749          | 1375.56              | 5                       |
| 2      | 305.000          | 1220.00              | 8.5                     |
| 3      | 280.251          | 1375.56              | 16.25                   |
| 4      | 305.000          | 1220.00              | 8.5                     |
| 5      | 340.000          | 1220.00              | 5                       |
| 6      | 305.000          | 1220.00              | 8.5                     |
| 7      | 280.251          | 1064.44              | 25.5                    |
| 8      | 305.000          | 1220.00              | 8.5                     |
| 9      | 270.000          | 1220.00              | 26                      |
| 10     | 305.000          | 1440.00              | 7.75                    |
| 11     | 305.000          | 1220.00              | 8.5                     |
| 12     | 305.000          | 1000.00              | 9.5                     |
| 13     | 329.749          | 1064.44              | 5.5                     |
In the composite cleaning system at 335 °C and 1440 W, the paint had been cleaned completely in 5 min, as shown in Figure 12b. Because the effect of ultrasonic power on the cleaning cycle was a little small, it would have little influence on the cleaning cycle only under the effect of pyrolysis and oxidation reactions. However, when the paint specimen was heated in air at 335 °C and 5 min, there was still large scale of paint on the part surface, as shown in Figure 12c. Some areas were gradually blackened and more holes appeared on the paint surface, indicating the occurrence of pyrolysis and oxidation reactions. Figure 13 shows that the total mass loss rate of paint in air was about 53%. The $T_g$ curve of the mixture showed that after 470 °C, the variation trend was consistent with the $T_g$ curve of salts, indicating that the paint was completely removed at 470 °C.

To investigate the reasons for the above differences, the Coats–Redfern integral method was used. In an infinitesimal time interval, the nonisothermal process can be regarded as an isothermal process. The decomposition rate equation of the paint could be expressed as

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \tag{1}$$

where $d\alpha/dt$ is the conversion rate, $\alpha$ is the transmission rate, $\alpha = (m_0 - m)/(m_0 - m_f)$, $m_0$ is the initial mass, $m$ is the mass at the time of $t$, $m_f$ is the final mass, $k(T)$ is the reaction rate constant, $f(\alpha)$ is the decomposition process reaction function, $t$ is the time, and $T$ is the thermodynamic temperature.

$k(T)$ could be expressed by the Arrhenius equation

$$k(T) = A\exp\left(\frac{-E}{RT}\right) \tag{2}$$

where $A$ is the preexponential factor ($\text{min}^{-1}$), $E$ is the activation energy ($\text{kJ/mol}$), and $R$ is the gas molar constant, whose value is $8.314 \times 10^{-3}$ kJ/(mol K). Since the experiment used the constant velocity heating method, the heating rate was $\beta = dT/dt$, so formula (1) could be converted into

$$\frac{d\alpha}{dT} = \frac{1}{\beta}k(T)f(\alpha) \tag{3}$$

By integrating formula (3) and assuming that the decomposition of the paint belonged to the first-order reaction, the decomposition process reaction function $f(\alpha)$ could be expressed as $f(\alpha) = (1 - \alpha)^n$, where $n = 1$, so formula (4) could be obtained

$$\ln \frac{-\ln(1 - \alpha)}{T^2} = -\frac{E}{RT} + \ln \left[\frac{AR}{\beta E(1 - 2RT/E)}\right] \tag{4}$$

Figure 11. Change of cleaning cycle with temperature and ultrasonic power.

Figure 12. Comparison of paint surface before and after heating at 300 °C and 5 min of cleaning time.

Figure 13. TG curve of paint, salt, and their mixture.
In this test, the range of \( T \) was 298–873 K, \( E \) was about 100 kJ/mol, \( R = 8.314 \times 10^{-3} \text{kJ/(mol K)} \), then \( 0 < 2RT/E \ll 1 \), so the \( \ln[(AR/(\beta E))(1−2RT/E)] \) in formula (4) was approximately constant. Through the test, the paint conversion rate at each temperature \( \alpha \) was obtained. Then, \( 1/T \) was plotted against \( \ln[-\ln(1−\alpha)/T^2] \) and the curves were linearly fitted. Therefore, the linear fit curves of 1 and 3 are shown in Figure 14.

![Figure 14. Linear fitting lines of paint and mixture.](image)

It can be seen that the curves were a straight line with high linear correlation. By fitting the slope and intercept of the curves, the activation energy \( E \) and the preexponential factor \( A \) could be calculated, respectively.

It can be inferred from Figure 14 that linear correlation coefficients \( (R^2) \) were 0.9976 and 0.9496, indicating that the test met the first-order reaction kinetics. The activation energy of paint alone was 114.4 kJ/mol. But the activation energy of paint in salt was just 74.1 kJ/mol, which was much lower than that of the paint alone. Therefore, salt could effectively reduce the activation energy required for the decomposition of paint and significantly improve the paint removal efficiency.

The reduction of activation energy was due to the fact that the NaNO₃–KNO₃–NaOH–NaNO₂ system could play a bridge role in the reaction process and provide a good dissolution environment for oxygen. The oxygen existed in the form of peroxide ions (\( O_2^− \)) and superoxide ions (\( O_2^− \)) in the cleaning system. Peroxide ions and superoxide ions were more oxidizing than the oxygen, which was the reason why paint more easily decomposed in the salt system. Nitrate ions could further promote the conversion of peroxide ions into superoxide ions, enhancing the ability of paint removal. So, the adhesion strength of paint would decrease. By their high fluidity, the paint fragments could easily peel off from the substrate and would be prevented from attaching to the substrate again.

5. CONCLUSIONS

In summary, molten salt ultrasonic composite cleaning had a good effect on paint removal. First, the mechanism of molten salt cleaning and the mechanism of ultrasonic cleaning were reviewed. To further describe the composite cleaning mechanism, the components and internal structure of paint were analyzed. Results showed that the paint had a significant layered structure. The total thickness was about 100 \( \mu \text{m} \), and the main components were organic matter, including ester groups, epoxy groups, and aromatic compounds. Then, combining with the thermal environment, the cleaning medium, and ultrasound, the composite cleaning mechanism was described in terms of three aspects: thermal effect, chemical reaction, and ultrasonic effect.

(1) Thermal effect. In a high-temperature environment, the difference of two materials’ expansion coefficient led to different thermal expansions, making the paint surface have apophysis and cracks. With low surface tension, the cleaning medium could easily combine with the paint surface and infiltrate into these cracks. Besides, about 3 \( \text{cP} \) of low viscosity made the medium keep high fluidity, preventing the paint fragments from attaching to the substrate again and enhancing the ion migration rate.

(2) Chemical reaction. Through analyzing the gaseous production, we could find that there were 18 kinds of compounds in complex gas, including seven kinds of hydrocarbons, five kinds of aldehydes, three kinds of esters, two kinds of ketones, and one kind of alcohol. Combining with the ions and the thermal environment, the chemical effect was mainly the effect of pyrolysis and oxidation reactions.

(3) Ultrasonic effect. The high-frequency oscillation enhanced the fluidity of medium, resulting in the uniform distribution of the internal temperature field and the ion concentration. Besides, the ultrasonic cavitation effect caused more cracks on the paint surface. Under the effect of shock generated by cavity bubbles, the paint surface suffered from fatigue damage. So, the paint fragments were easily separated from the substrate.

The cleaning experiment showed that the effect of temperature on the cleaning cycle was higher than that of ultrasonic power. But when paint was heated in air, which also had the thermal effect and the reactions of pyrolysis and oxidation, the cleaning results were very poor. The reason was that salt could reduce the reaction’s activation energy from 114.4 kJ/mol of paint alone to 74.1 kJ/mol. Because of the limited dimensions of the cleaning tank, the dimensions of the cleaning objective must be suitable for the tank. So, it is very vital to select an appropriate cleaning tank for mechanical parts.

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**Notes**

The authors declare no competing financial interest.

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