Plasmonic and Superhydrophobic Self-Decontaminating N95 Respirators

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ABSTRACT: The COVID-19 pandemic is endangering the world due to the spread of respiration droplets with viruses. Medical workers and frontline staff need to wear respirators to protect themselves from breathing in the virus-containing respiration droplets. The most frequently used state-of-the-art respirators are of N95 standard; however, they lack self-decontamination capabilities. In addition, the viruses and bacteria can accumulate on the respirator surfaces, possessing high risks to the wearers over long-term usage. Photothermal decontamination is a contactless, fast, low-cost, and widely available method, capable of decontaminating the respirators. Herein, we report a plasmonic photothermal and superhydrophobic coating on N95 respirators, possessing significantly better protection than existing personal protection equipment. The plasmonic heating can raise the surface temperature to over 80 °C for this type of respirator within 1 min of sunlight illumination. The superhydrophobic features prohibit respiration droplets from accumulating on the respirator surfaces. The presence of the silver nanoparticles can provide additional protection via the silver ion’s disinfection toward microbes. These synergistic features of the composite coatings provide the N95 respirator with better protection and can inspire experts from interdisciplinary fields to develop better personal protection equipment to fight the COVID-19 pandemic.

KEYWORDS: COVID-19, plasmonic, superhydrophobic, photothermal, laser precision manufacturing

The COVID-19 outbreak infected over 3 million people worldwide by April 2020. This disease is spreading by the coronavirus within respiration droplets or nasal secretions, which can enter the human body through the nose, eyes, and mouth. Although wearing goggles can protect the wearer from the virus entering through the eyes, respirators are needed to cover the nose and mouth for medical workers. The N95 respirators are made of polymer fibers, such as polypropylene, and although they possess hydrophobic surfaces, aqueous respiration droplets can still stay on these fibers. So the virus, bacteria, and other microbes can remain and grow on the surfaces of these respirators, forcing the wearers to replace these respirators regularly. However, many medical workers and frontline staff have to reuse these unsafe respirators or wear them for a quite a long time due to the global shortages of personal protection equipment and the urgent requirement to take care of large numbers of patients. Developing respirators that can be worn long-term is in extremely high demand, especially under the current COVID-19 pandemic.

Decontamination methods for N95 respirators have been emerging research and developed since the COVID-19 outbreak. Chemical- and heat-based decontamination are the major methods to sterilize N95 respirators. Directly using high-concentration ethanol for decontamination is not recommended, since the ethanol might break down the polypropylene-based fibers. Vapor phase hydrogen peroxide has been studied for decontamination of N95 respirators, and significant reduction of bacterial loads has been demonstrated without affecting the filtration efficiency or the fit to the wearer. However, the availability of H2O2 might be affected by the lockdown and logistic shortages, and wide adoption of this method for decontaminating N95 respirators in hospitals and
other public sectors is still challenging. In addition, the heating method can also effectively deactivate the severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2) viruses, as shown in Figure 1a, due to the instability of their spike proteins and RNA at temperatures over 70 °C. Cui’s group recently studied the stability of different brands of N95 respirators under heating at different humidity levels. They found that pristine N95 respirators showed no degradation over 10 cycles of treatment at 85 °C and a less than 4% degradation after 20 cycles of heating treatment. The 5 cycles of hot air treatment for N95 respirators only showed 1.5% change in fit factor, according to the OSHA protocol fit test. So the heating method is effective for reusing the N95 respirators without damaging the integrity or the fit for wearers. However, the existing heating methods for decontamination of N95 respirators need electrical or other thermal...
sources to heat the samples. Alternative contactless, easily accessed, and low-cost methods are preferable for promoting the effective decontamination of N95 respirators worldwide, especially for developing countries where the medical systems are fragile.

Plasmonic heating is an important method for converting photonic energy to heat, through the vibration of photon-exited electrons into phonons. Metallic nanoparticles with ultrathin features possess strong absorption of solar energy through these size-dependent plasmonic effects. In addition, the metal ions also show strong antibacterial effects, especially the silver and copper elements. Silver nanoparticles have been widely used for wound dressing materials, medical devices, and textiles due to their bactericidal effects. It has been found that the ion release rates and the antibacterial performance depended on the size of the silver nanoparticles. Herein, we use laser-induced forward transfer to deposit silver nanoparticles with wide size distributions onto N95 respirators. The plasmonic absorption and silver ions on these N95 respirators can provide better protection against microbes. In addition, by fine-tuning the laser parameters, additional laser-induced graphene can be deposited together with the silver nanoparticles, permitting enhanced photothermal absorption and superhydrophobic surfaces. These synergetic effects can provide better long-term protection against respiration droplets containing the virus and bacteria.

RESULTS AND DISCUSSIONS

The fabrication process of silver coating on N95 respirators is shown in Figure 1. The thin silver film is deposited on the commercial polyimide film using sputtering at a 10⁻⁶ Torr vacuum, with controlled thickness monitored by an in-chamber balance. The silver nanoparticles can then be deposited onto the N95 directly via pulse mode laser-induced forward transfer as method 1 (M1), as shown in Figure 1b. The whole manufacturing process can be scaled up to roll-to-roll laser manufacturing, as illustrated in Figure 1c. Method 2 (M2) includes an additional step for transforming the silver nanoparticle onto another polyimide film via continuous wave (CW) mode laser-induced forward transfer. Then the deposited silver nanoparticles and the generated graphene are further deposited onto the N95 respirator via pulsed mode laser-induced forward transfer, as shown in Figure 1d. The appearance of the M2 N95 respirator is shown in Figure S1. M1 is only Ag coated in order to analyze the plasmonic effect, while M2 is a revised approach to improve the macroscaled photothermal and hydrophobicity properties. Due to the high-speed laser scanning process, these two steps are both compatible with the roll-to-roll production, as illustrated in Figure 1e. Therefore, these two methods both possess high scalability for automatic mass production, which can be easily integrated into the modern respirators’ production lines.

To analyze the microstructures of the deposited silver nanoparticles, the surface coating materials on the N95 respirators were collected and characterized using electron microscopy. A typical morphology of the M1 sample from a 50
nm thick precursor is shown in the field emission scanning electron microscopy (FESEM) images, as in Figure 2a. The bright-colored silver nanoclusters with sizes up to several hundreds of nanometers were distributed on the surfaces of the nonwoven fibers of the N95 respirators. The chemical compositions were identified to be silver, according to the energy dispersive X-ray (EDX) analysis in Figure S2. The high-resolution transmission electron microscopy (HRTEM) images clearly show the lattice structures of the M1 silver nanoparticles, presented in Figure 2b. The black spheres were silver nanoparticles, with a size distribution from 5 to 50 nm, as presented in Figure S3a. The wide size distributions of the observed nanoparticles, in the inset of Figure 2b, are the results of the Gaussian shape energy distribution of the laser beam. Furthermore, as the thickness of the precursor film increased, the size distributions tended to be wider, as shown in Figure S4, which is due to the growth of the grain when the temperature increases in the manufacturing process. In contrast, for the CW mode laser synthesized samples from M2 with a 50 nm thick silver film precursor, obvious differences of its morphology were observed in the FESEM images, as shown in Figure 2c. The size of the nanoparticles in the M2 samples was evenly distributed with significantly small variance, as shown in Figure S3b. And the background from M2 was filled with porous graphene composites, as verified by the D, G, and 2D peaks in the Raman characterization, as obtained in Figure S5. According to the EDX analysis in Figure S6, the carbon atomic ratio within the compositions was significantly increased. Similar crystalline structures were observed for the M2 silver nanoparticles in Figure 2d. Power allocations of different laser modes and the use of polyimide resulted in a smaller variance for the particles size manufactured by M2 than M1, as indicated in the inset figure. The sizes of the silver nanoparticles, which is demonstrated in Figure S7, also increased as the thickness of the precursor increased. So the thickness of the precursor silver thin film and laser parameters played a significant role in determining the final morphology and sizes of the plasmonic silver nanoparticles on N95 respirators.
The crystalline structures of the silver nanoparticles deposited from the different modes were studied by X-ray diffraction (XRD). As shown in Figure 3a, both the M1 and M2 mode silver showed face-centered cubic (fcc) crystalline structures with (111), (200), (220), and (311) identifying peaks. As the thickness of the precursor silver thin films increased, the intensities of the XRD peaks tended to increase for both the M1 and M2 approaches, as shown in Figure S8a, indicating that the amounts of silver nanoparticles on the samples increased. Meanwhile, the half-width as measured in Figure S8b and c was not significantly increased, which pointed out the crystallinity was not influenced when more silver nanoparticles were present, and it was consistent with the FESEM and HRTEM images in Figure 2.

To study the plasmonic effects of the synthesized silver nanoparticles, the optical absorption spectra were measured by a spectrometer from 300 to 2500 nm with an integration sphere, as shown in Figure 3b. The absorption spectra of the M1 sample (blue curve) showed an obvious absorption peak near 405 nm compared to the pristine sample (green curve), indicating the plasmonic-enhanced absorption of the silver nanoparticles. Meanwhile, the M2 mode included graphene in the samples and resulted in a significantly higher absorption of over 95% across the whole spectrum, which can contribute to the significantly high photothermal effect using broadband light sources.

The introduction of graphene can also contribute to the superhydrophobic surfaces on the N95 respirators. The surface energies of the pristine M1 and M2 coated N95 respirators were estimated by water contact angle measurements. The pristine N95 respirators showed a contact angle of 116°, as shown in Figure 3c, indicating its hydrophobic state. The M1 coated respirators showed a 68° contact angle, indicating that the pulsed laser printed silver would render the surfaces of the N95 respirator hydrophilic, as shown in Figure 3d. At the same time, the coated N95 respirator from M2 demonstrated superhydrophobic features, with a static contact angle over 140°, as shown in Figure 3e. The whole N95 respirator coated with M2 by this method also exhibits an excellent superhydrophobic performance (Supplementary Video 1). The water droplet will attach on the pristine N95 surfaces. In contrast, the water droplet will freely roll off on the laser-coated superhydrophobic N95 surfaces, which can provide better protection from the SARS-CoV-2 viruses. Besides the outstanding optical absorption features, the dual-mode M2 coating method provides better protection from the aqueous respiration droplets, while the M1 coating can decrease the hydrophobic state of the N95 respirator, which might lower its protection functions.

Due to the plasmonic-enhanced absorption, selective absorption enhancement can be utilized by a laser sterilization process. A 405 nm laser diode was chosen for heating the silver-coated respirator, due to the plasmonic-enhanced absorption peak at this wavelength, as shown in Figure 3b. The laser diode can focus on the surface of the coated respirator for decontamination, as illustrated in Figure 4a. By moving the laser diode positions relative to the N95 respirator, the laser can scan all the areas of the respirator at a fast speed. The localized laser illumination can excite the plasmonic vibration around the silver nanoparticles and provide strong heating of the surrounding area, as illustrated in the inset of Figure 4b. By optimizing the laser power and the laser speed, a Gaussian-shaped thermal profile can be created using the laser illumination, which is measured by the infrared camera, as shown in Figure 4b for the M2 sample and Figure S9 for the M1 sample. The width of 1 mm with a temperature over 60 °C can be realized at a 100 mm/min scanning speed, with the highest temperature over 80 °C, which is far higher than the pristine N95 respirator as a control sample with a temperature lower than 40 °C. The fast scanning speed can quickly scan the entire area (∼0.02 m²) of the N95 respirator. After all the areas are heated, all the potential remaining viruses can be inactivated. The long-term laser decontamination stabilities were also tested. With laser heating temperatures over 85 °C, the microstructures did not show significant degradation, up to 100 cycles of 405 nm laser decontamination, as shown in Figure 4c. The contact angle measurement also revealed that the superhydrophobic features can be maintained after 100 cycles of laser decontamination, as shown in Figure 4d. However, when the laser temperature was over 125 °C, some breakdown of the fiber was noticeable, as shown in Figure S10. So it is essential to control the laser decontamination temperature to preserve the integrity of the N95 respirator.

The solar photothermal effects of the pristine and M1 and M2 coated N95 respirators were analyzed by illuminating with a solar simulator using an AM 1.5 filter. The temperature of the pristine N95 respirator was plotted against time, as shown in the blue diagram in Figure 5a. After 5 min of solar illumination, the temperature of the pristine respirators was stabilized at 40 °C. For the silver-nanoparticle-coated M1 sample, a slight
increase in the temperature was observed, indicating localized plasmonic heating effects, as shown in the green plot in Figure 5a. Meanwhile, the M2 sample shown in the red curve displayed a significant heating effect compared with the previous samples. The surface temperature of the M2 sample quickly jumped to over 60 °C in 10 s and stabilized over 80 °C after 60 s of solar illumination at 1000 W/m². This high temperature can provide strong decontamination toward the SARS-CoV-2 viruses. The result of the thermal conductivity for the M2 samples in Figure S11 and the backside temperature of the mask after 10 min of 1 sun solar illumination in Figure S12 ensured the feasibility for daily use. Moreover, an outdoor test was carried out on a cloudy day on April 26, 2020, in Hong Kong, as shown in Figure S13a. The surface temperature of the respirator rose to over 60 °C, which is consistent with the indoor experimental result using a solar simulator at around 600 W/m², as demonstrated in Figure S13b. So the additional participation of laser-induced graphene in the M2 samples can boost the photothermal performance for heating the N95 respirator surfaces.

To evaluate the structural and surface energy stabilities of the M2 respirators over long-term solar heating decontamination, the samples were illuminated under 1 sun intensity for 72 h aging tests. The microstructures of the M2 coating did not show significant change before and after the 72 h aging test, as shown in Figure 5b. The water contact angle of the coated respirator after the 72 h aging demonstrated a similar superhydrophobic effect, as shown in Figure 5c. The above results indicate that the M2 coated N95 respirator can work stably during long-term solar heating.

The N95 respirators have been successfully coated with plasmonic silver nanoparticles and laser-induced graphene via laser-induced forward transfer. The size distributions of the silver nanoparticles were synergistically determined by the sputtering conditions and the laser writing processes. The size distributions of the sputtered thin films had been widely studied, where randomness of the grain sizes was contributed by the random ion sizes for depositing the nanoparticles. At the same time, the laser parameter also played an important role in determining the final distributions of the nanoparticles. For laser ablation with low energy, the polyimide film only suffered from mechanical deformation. When the laser ablation was large enough, rupture occurred on the polyimide surface and led to cracking with more violent transfer with splashing on the flat surfaces, which would result in a wider size distribution. In the current study, the parameters of the precursor and laser writing significantly influenced the size distributions of the silver nanoparticles. For increased precursor silver thin film thickness, a wider size distribution was observed, as verified by FESEM and HRTEM characterizations in Figure 2.

The amounts of nanoparticles were also increased, according to the intensity of the XRD and electron microscopy characterizations. Meanwhile, the increased laser power can also result in a higher amount of silver nanoparticles, as presented in the XRD in Figure S8. Since the different nanoparticle sizes can result in different plasmonic vibration modes, as shown in the optical absorption of composites deposited onto glass in Figure S15, it is possible to tune the plasmonic-enhanced absorption wavelength by controlling the precursor thickness and laser writing parameters. The resulting enhanced plasmonic absorption near 405 nm can be utilized for heating the N95 respirator using a low-cost laser diode at 405 nm (around $1 USD each). By tuning the 405 nm laser intensity and scanning speed, we can control the resulting plasmonic heating with the temperature ranging from 60 to 125 °C. The strong photothermal effect of the plasmonic respirators can decontaminate the N95. However, overheating can result in a breakdown of the nonwoven fibers when the photothermal temperature is over 125 °C. At the same time, laser-induced graphene can be added to the silver composite by adding a CW mode laser-induced forward transfer. The addition of few-layer graphene can provide a superhydrophobic surface and increased broad photon absorption. The superhydrophobic coating can prevent the attachment of respiration droplets on the respirator surfaces, mimicking the lotus leaf self-cleaning phenomena. The graphene-enhanced optical absorption also increases the photothermal performance. Under solar illumination at 1000 W/m², the surface temperature of the N95 respirator can be increased over 80 °C after 1 min. The native thermal conductivity of the N95 respirator was measured to be 0.04 W/m-K, enabling the localized heating of the plasmonic silver composite on the surfaces. This high temperature is sufficient to inactivate the spike protein of the SARS-CoV-2 virus. The SARS-CoV-2 virus can only enter human cells through the angiotensin-converting enzyme 2 (ACE2) receptor, so this photothermal decontamination method can provide effective protection to the wearer against these viruses under sunlight.

This laser-induced forward transfer method preserves the original filtration features of the N95 respirators. Although direct laser-induced forward transfer using the continuous wave mode can also transfer the graphene onto other high-melting-temperature substrates, the high temperature during the transfer would melt the nonwoven fibers in the N95 respirators. So the low temperatures of the second step pulse-mode laser-induced forward transfer for the final deposition on these fibers in M2 are critical for preserving the original filtration performance of the N95 respirator. Compared to directly sputtering silver nanoparticles onto the respirators, the laser printing method is more production compatible with the roll-to-roll process. In addition, the high temperature during sputtering might melt the fibers of the masks, especially with the vacuum chambers. Compared to laser-induced graphene with CO₂ lasers, this direct printing method can avoid the additional laminating process for transferring a plasmonic silver and graphene composite onto the N95 respirators. Due to the weighting distribution of silver particles from the outer surface to the second layer as shown in Figure S16 and illustrated in Figure S17, the Ag coating on the second layer, where less graphene and solar illumination is present, provides additional protection through plasmonic heating. In addition, with the help of broadband absorption enhancement by graphene on the first layer, this plasmonic composite coating results in synergistic protection of the wearers. Although M1 mode N95 is easier to fabricate compared to the M2 mode, the loss of hydrophobic features would result in higher chances for the incoming droplets to attach on the surfaces of nonwoven fibers. In comparison, the M2 mode equips the N95 respirators with superhydrophobic properties through the addition of graphene nanostructures. The incoming respiration droplets would have fewer opportunities to stay on the silver/graphene-coated N95 surface, due to the lotus-leaf-mimicking superhydrophobic surfaces.

The use of silver nanoparticles for N95 respirator coating is crucial for both the plasmonic heating and antimicrobial
considerations. \(^3^9\) Laser-induced graphene is biocompatible as examined with zebrafish during development. \(^4^0\) However, the photothermal effect introduced by the silver nanoparticles with either laser diode decontamination or solar illumination can inactivate the possible remaining SARS-CoV-2 viruses. In addition, other microbes such as bacteria, which might also pollute the respirator, can also be sterilized through the silver ion release. \(^4^1\) Meanwhile, we had experimentally verified the antibacterial properties of laser-induced forward transferred graphene through the synergistic photothermal and super-hydrophobic effect using glass substrates. \(^4^2\) And this solvent-free strategy is significantly greener compared to other wet chemistry methods. So this laser-induced forward transfer method for deposition of plasmonic composites can offer better protection against the COVID-19 pandemic and can provide significantly advanced approaches compared to existing methods for coating plasmonic materials.

Besides the better protection compared to the pristine N95 respirator, the laser-induced forward transferred strategies also have other advantages. Due to the flexible nature of the precursor materials with silver and polyimide, the whole laser manufacturing processes can easily be integrated with roll-to-roll production lines, which is compatible with existing respirator production methods. The production rates of the laser printing can also be increased by using higher power lasers and faster galvo scanning systems with outstanding scalability for mass production. In addition, the raw material cost of polyimide is low, at around $0.1 USD per N95 respirator. Although the use of silver seems expensive, the use of only a 50 nm thickness of silver significantly lowers the materials cost down to $0.05 USD per N95 respirator. So this laser-printed silver composite N95 respirator is economical for actual implementation. Although quarantine and lockdown can effectively lower the risk of spread of SARS-CoV-2 virus, societies suffer a lot due to the halt of normal activities, including scientific research and development. The wide adoption of respirators with enhanced protection can help lower the risks of spreading COVID-19 when gradually restoring normal activities.

At the same time, plasmonic photothermal decontamination can be realized using only solar energy. And a surface temperature of 60 °C can be achieved with a solar intensity of 600 W/m², indicating that the solar energy in most areas of the Earth is sufficient for photothermal decontamination of viruses and bacteria. Furthermore, the medical and decontamination resources in Africa are unfortunately more fragile than in developed countries. The use of the abundant solar energy in these areas for decontamination of respirators is practical and meaningful for combating COVID-19 with great sociological impact.

However, there are some limitations in the current study. Although the high temperature can provide sufficient inactivation for the SARS-CoV-2 virus, \(^2^4\) clinical testing for the actual decontamination has not been presented. Due to the large numbers of N95 respirators, the decontamination process of relatively short duration can save considerable time for medical workers and frontline staff. At the same time, some commercially available high-power GaN LEDs also had similar illumination ranges near 405 nm, which can also be utilized for photothermal decontamination, which can provide faster decontamination compared to laser scanning. The photothermal decontamination with these LEDs is worthy for future study. In addition, some other plasmonic alloys can provide better optical absorption compared to silver, which should be studied in the near future. The mechanical stability of the coating can be further enhanced by adding longer-chain polymer materials in future studies. It can be envisioned that there will be more advanced plasmonic antiviral methods developed by the broad scientific community to fight against the COVID-19 pandemic.

CONCLUSIONS

The laser-induced forward transfer of plasmonic composite materials on the N95 respirator was studied. The optical absorption enhancement using a 405 nm laser diode can be realized by choosing optimized parameters for a precursor thin film and laser printing parameters. The functionalized N95 respirator showed excellent superhydrophobic and photothermal performances alongside the silver ion release toward microbes in respiration droplets, synergistically providing better protection against the SARS-CoV-2 virus. This work will inspire the scientific and engineering community to develop better applications with their expertise in fighting the pandemic now and in the future. \(^4^2\)

METHODS

Fabrication. The 3M 8201 N95 respirators and 100 μm thick polyimide films were used after purchase without any treatment. The silver thin film was deposited on the polyimide with a Denton Discovery 8 sputtering system, with a typical thickness of 50 nm as monitored in the in-chamber balance monitor. Both the CW and pulse mode scribing were conducted by a DMG Lasertec 40 laser machining system with 1064 nm wavelength and 20 μm spot size. M1 adopted only the one-step pulse laser mode at 10 ns for the transfer. The silver particles were directly transferred to the N95 respirator using a 0.5 W power and 400 mm/s speed. The M2 strategy used the CW laser mode with a 3 W power and 400 mm/s scan speed, to transfer the silver to the polyimide acceptor. The laser-induced graphene was also generated and transferred to the acceptor polyimide film during this high-temperature step. \(^3^9,^4^3,^4^4\) Later the silver and graphene flakes in the acceptor were deposited onto the N95 respirator by the 10 ns pulse mode laser at 0.5 W power and 400 mm/s speed.

Characterization. The images of the microstructures of the respirators were acquired by a Tescan MAIA3 FESEM. A Sindatek 100SB optical contact angle meter was applied to measure the surface energies of the sample with a 10 μL water droplet. The Raman spectra were carried out by a WITEC confocal Raman system, using a 532 nm laser source. To characterize the photothermal properties, a Newport 91160 solar simulator with an AM 1.5 laser source. To characterize the photothermal properties, a Newport 91160 solar simulator with an AM 1.5 filter was used, with light intensity calibrated at 1000 or 600 W/m², respectively. The laser decontamination was carried out using a homemade laser diode system (total cost less than $100 USD) with motors controlled by an Arduino microcontroller, as reported previously. \(^4^5\) The optical absorption spectrum was collected by a Hitachi UH4150 spectrophotometer equipped with an integrating sphere. A Fluke Ti200 infrared camera was used to measure the surface temperatures of the coated respirators. The thermal conductivity was studied by an Anter Flashline 2000 thermal conductivity analyzer.

ASSOCIATED CONTENT

\* Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsnano.0c03504.

Additional information (PDF)

Comparison of the wetting properties between the pristine N95 surface and plasmonic N95 surface (MP4)
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Notes
The authors declare no competing financial interest.

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