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## **Fly-through synthesis of nanoparticles on textile and paper substrates**

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### **Keywords:**

High temperature, Radiative heating, Nanoparticle synthesis, Temperature-sensitive substrates, Roll-to-roll

**Abstract**

The fast and efficient synthesis of nanoparticles on flexible and lightweight substrates is increasingly critical for various medical and wearable applications. However, conventional high temperature (high-T) processes for nanoparticle synthesis are intrinsically incompatible with temperature-sensitive substrates, including textiles and paper (i.e. low-T substrates). In this work, we report a non-contact, ‘fly-through’ method to synthesize nanoparticles on low-T substrates by rapid radiative heating under short timescales. As a demonstration, textile substrates loaded with platinum (Pt) salt precursor are rapidly heated and quenched as they move across a 2000 K heating source at a continuous production speed of  $0.5 \text{ cm s}^{-1}$ . The rapid radiative heating method induces the thermal decomposition of various precursor salts and nanoparticle formation, while the short duration ensures negligible change to the respective low-T substrate along with greatly improved production efficiency. The reported method can be generally applied to the synthesis of metal nanoparticles (e.g. gold and ruthenium) on various low-T substrates (e.g. paper). The non-contact and continuous ‘fly-through’ synthesis offers a robust and efficient way to synthesize supported nanoparticles on flexible and lightweight substrates. It is also promising for ultrafast and roll-to-roll manufacturing to enable viable applications.

## Introduction

Supported nanoparticles are widely used in the fields of plasmonic, catalysis, energy storage, antibiosis, and energy conversion.<sup>1-5</sup> The fast and efficient synthesis of supported nanoparticles is in urgent demand to promote their viable applications.<sup>6-10</sup> Conventionally, high-temperature (high-T) methods, such as annealing, thermal reduction and calcination, offer favorable thermodynamic environments for efficient and controlled nanoparticle synthesis.<sup>11-13</sup> In addition, high-T methods with a shorter duration time, such as microwave radiation as well as flame synthesis methods, can produce supported nanoparticles within several minutes or even seconds.<sup>14-22</sup> These simple and physical deposition methods based on high temperature processes are easily scalable and sustainable with minimal chemical pollutants and solution waste, and are favorable for efficient synthesis of supported nanoparticles.

However, these methods heat the entire sample to high temperatures and have seldom been demonstrated for substrates with low thermal stability, such as textiles and paper (i.e. low-T substrates), as the structure of these substrates is easily compromised in the heating process. Yet, nanoparticles dispersed on flexible and lightweight low-T substrates are increasingly critical for various medical and wearable applications (e.g. antibacterial surfaces, disease detection, and bio-sensing), where large-scale and low-cost products are used.<sup>23-26</sup> These medical and wearable applications require a facile, scalable particle synthesis method that is also compatible with low-T substrates.<sup>27-30</sup> Therefore, it is crucial to overcome the dichotomy between ‘efficient high-T synthesis’ and ‘sensitive low-T substrates’ to enable the rapid synthesis of nanoparticles on flexible substrates in a scalable and sustainable manner.

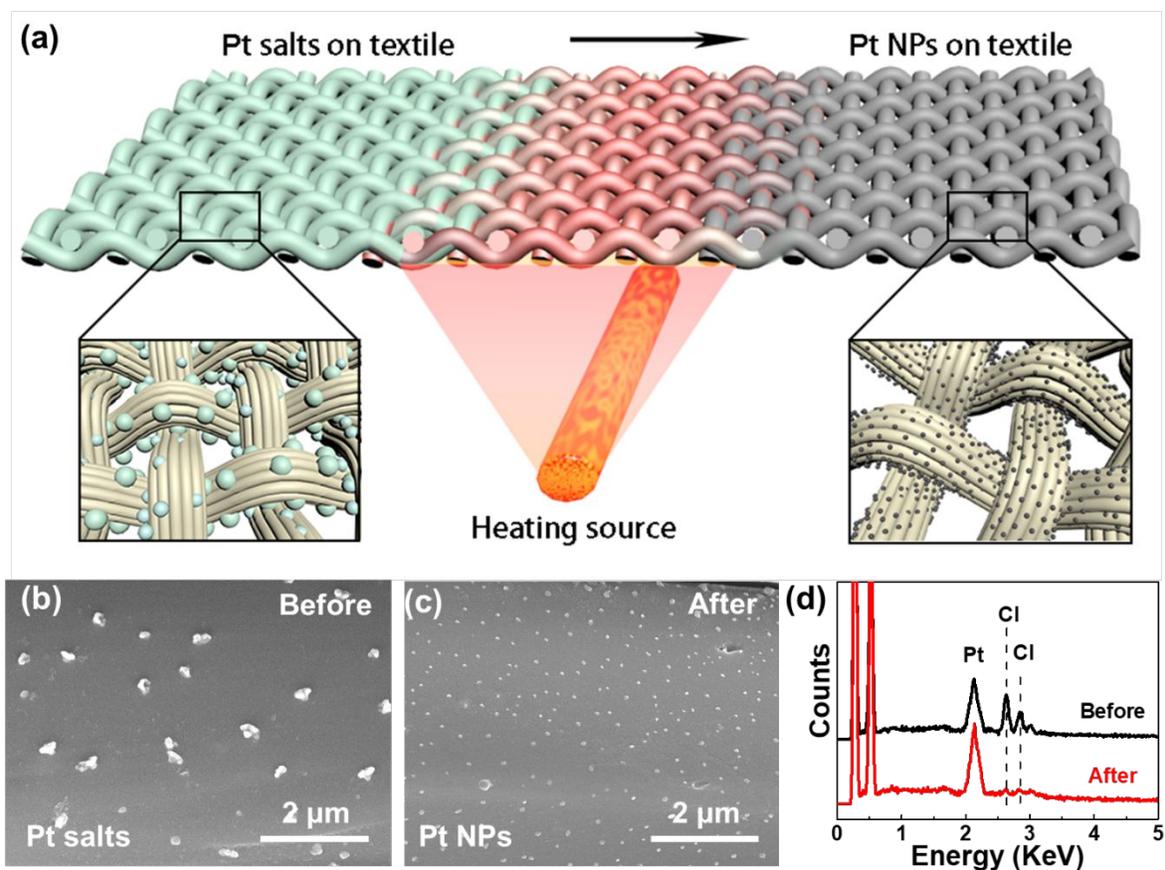
For the first time, we report a one-step, ‘fly-through’ method to synthesize well-dispersed nanoparticles on low-T substrates (i.e. textiles and paper) *via* a high-temperature, rapid radiative

heating. In a typical process, the metal precursor loaded textile is pulled across the top (0.5 cm) of the high-T heating source (2000 K) at a constant rate of  $0.5 \text{ cm s}^{-1}$ , after which the metal nanoparticles are uniformly dispersed on the textile. The high-temperature source enables the decomposition of the precursor salts and nanoparticle nucleation on the surface of the textile. In addition, the continuous ‘fly-through’ synthesis with limited short heating duration only leads to uniform nanoparticle formation while avoiding deterioration of the textile substrate. The non-destructive ‘fly-through’ method is generally applicable for the synthesis of other metal nanoparticles (e.g. Au and Ru) on various low-T substrates (e.g. paper). The non-contact and continuous particle synthesis is deemed compatible with roll-to-roll processes for scalable and ultrafast nanomanufacturing.

## Results and Discussions

The one-step, rapid radiative heating process is depicted in **Figure 1a**. The  $\text{H}_2\text{PtCl}_6$  precursor loaded textile is pulled across the high-T heating source quickly to induce nanoparticle formation by high-temperature radiation and subsequent quenching. The high intensity of the thermal radiation leads to the decomposition of the  $\text{H}_2\text{PtCl}_6$  precursor and transformation into Pt nanoparticles on the surface of the textile in several seconds. Owing to the short duration of the high temperature treatment process, the structure of the textile maintains largely intact and avoids high-temperature induced significant degradation. Therefore, the radiative heating method is uniquely suited to synthesizing metal nanoparticles on low-T textiles by limiting the high temperature duration and is similarly conducive to promote the production efficiency compared to conventional longtime heating nanoparticle synthesis methods.

To load the salt precursor, the textiles were soaked in  $\text{H}_2\text{PtCl}_6$  precursor solution ( $0.05 \text{ mol L}^{-1} \text{ H}_2\text{PtCl}_6$  in ethanol) for 20 min before drying in air for 1 h. The SEM images in Figure S1a-b show the crosswise aligned fibers loaded with  $\text{H}_2\text{PtCl}_6$  precursor, and the enlarged SEM image in Figure 1b reveals that the 200 - 400 nm salt crystals are sparsely dispersed on the surface of the textile fibers. In contrast, after the radiative heating process, numerous Pt nanoparticles are uniformly distributed on the textile with particle sizes between 60 - 140 nm (Figure 1c). Simultaneously, the surfaces of the fibers are largely unperturbed after the rapid radiative heating method and retain their configuration. The  $\text{H}_2\text{PtCl}_6$  precursor decomposition is further verified by the energy dispersive spectrum (EDS) displayed in Figure 1d. The chloride (Cl) peak decreases substantially in samples after the radiative heating treatment, indicating that most of the Cl element (in Pt salt) is gone after thermal decomposition and leaves metallic Pt nanoparticles. The synthesized Pt nanoparticles were also characterized by XRD (Figure S2) and TEM (Figure S3), confirming the particle size and crystalline metallic structure.<sup>31</sup> Therefore, by rapid radiative heating, precursor salt can decompose under a short duration thermal radiation and nucleate into metal nanoparticles without significant alteration to the textile fibers.



**Figure 1.** (a) Schematic of the ‘fly-through’ synthesis of Pt nanoparticles on textiles by the rapid radiative heating method. The  $\text{H}_2\text{PtCl}_6$  precursor loaded textile is pulled across the high-T heating source at a continuous rate of  $0.5 \text{ cm s}^{-1}$ . The radiation from the high-temperature source induces decomposition of salt precursor and nucleates Pt nanoparticles on the surface of the textile while keeping the low-T substrate intact due to the short heating duration. The morphology of the textile with  $\text{H}_2\text{PtCl}_6$  precursor (b) before and (c) after radiative heating. (d) The EDS spectrum of the textile with  $\text{H}_2\text{PtCl}_6$  precursor before and after radiative heating.

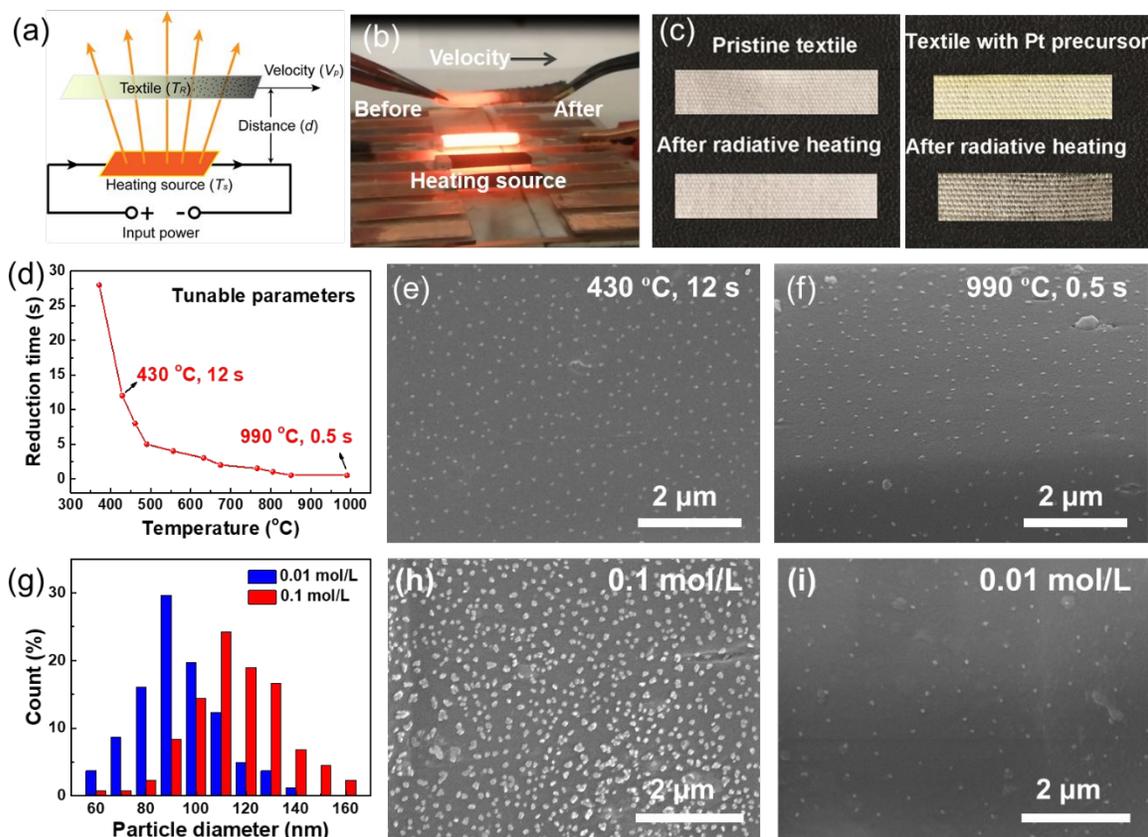
The tunable parameters of the radiative heating method for supported nanoparticles production are introduced in **Figure 2**, showing the ‘fly-through’ process as a flexible yet robust synthesis method. Figure 2a illustrates the setup of the rapid radiative heating method and the processing parameters including the radiative source temperatures ( $T_s$ ), the working distance between the

heating source and low-T substrate ( $d$ ), and the moving/production speed ( $V_p$ ). In our experiment, carbon nanofiber (CNF) films are used as radiative heating sources, which can be heated up to 3000 K by electrical joule heating and controlled easily by adjusting the electrical input.<sup>32</sup> Figure 2b shows the  $\text{H}_2\text{PtCl}_6$  precursor loaded textile being pulled across the radiative heating zone ( $T_s \sim 2000$  K, Figure S4a), 0.5 cm from the emissive source, at a velocity of  $0.5 \text{ cm s}^{-1}$ . The surface of the textile noticeably changes black after moving through high temperature heating source while the rest of textile is still a pristine white color. Control tests on pristine textiles without salt precursor show no change in color or morphology after radiative heating (Figure 2c), thereby indicating that the rapid heating does not induce significant decomposition or carbonization of the textile substrate. To reiterate, the color of the precursor-loaded textile changes from yellow (with  $\text{H}_2\text{PtCl}_6$  precursor) to black tint, which can be attributed to the precursor decomposition and nanoparticle formation (Figure 2c, right). The black tint is the result of light absorption by the as-formed Pt nanoparticles owing to the plasmonic effect.<sup>33,34</sup> Consequently, the color change in the textile is an important indicator of nanoparticle formation on textile during the radiative heating process.

The rapid radiative heating process can be easily tuned for the synthesis of supported nanoparticles. The temperature for the radiative heating ( $T_R$ ) is important for the decomposition of the precursor salts and it can be adjusted by the electrical input (Figure S4b) and working distance (Figure S4c) between 350 and 1000 °C. According to each specific  $T_R$ , there is a corresponding particle formation time as identified according to the color change on the textile, and confirmed through ex-situ measurements. Higher radiative temperatures require shorter radiative heating duration in the production process, as shown in Figure 2d. Although the synthesis temperature varies significantly, surprisingly, similar Pt nanoparticle distributions on the textile are shown in

Figure 2e and 2f, which are radiative heated at 430 °C for 12 s and 990 °C for 0.5 s, respectively. This indicates that the rapid radiative heating process renders similar size distributions as long as the salt precursor is completely decomposed. Therefore, the processing parameters in the radiative heating process can be easily adjusted for similar nanoparticles synthesis on textile substrates, thereby demonstrating its flexible and robust production capability.

In addition, by tuning the initial salt concentration on the textile material, the particle size and distribution can be adjusted. As demonstrated in Figure 2g, soaking the textile in  $\text{H}_2\text{PtCl}_6$  precursor solution with 0.1 mol L<sup>-1</sup> and 0.01 mol L<sup>-1</sup> concentrations result in different particle size distributions centered around 110 nm and 90 nm nanoparticles, respectively, according to the SEM images in Figure 2h and 2i. The high concentration specimen (0.1 mol L<sup>-1</sup>) shows a dense scattering of Pt nanoparticles on the surface of the textile fibers after radiative heating. In contrast, the low concentration specimen (0.01 mol L<sup>-1</sup>) demonstrated a sparse scattering of nanoparticles that are ~20 nm smaller on average (Figure 2i). Therefore, the particle diameter and loading density of the supported nanoparticles on the textile are tunable through adjustments to the initial salt loading.

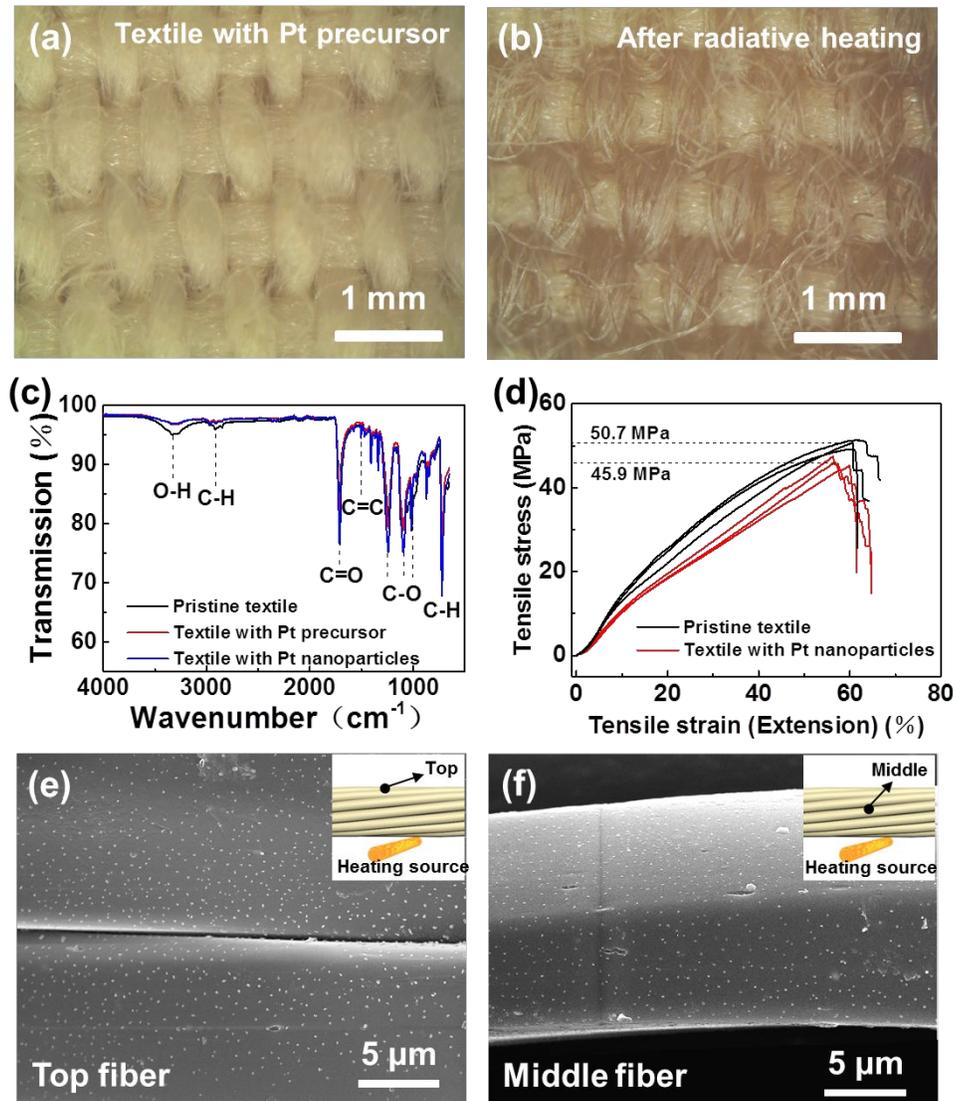


**Figure 2.** Kinetic control in the rapid radiative heating process. (a) A side-view schematic detailing the pertinent parameters of the radiative heating method. (b) The corresponding side-view image of the radiative heating process in which a precursor-loaded textile is pulled across the emissive heating source. (c) Photographs of a pristine and precursor-loaded textile before and after undergoing the radiative heating method, respectively. (d) The relationship between the temperature and the reduction time for the Pt nanoparticle formation. The morphology of the Pt nanoparticles on the textile through (e) radiative heating at 430 °C for 12 s and (f) radiative heating at 990 °C for 0.5 s. (g) The size distribution of the Pt nanoparticles on textiles soaked in 0.1 mol L<sup>-1</sup> and 0.01 mol L<sup>-1</sup> precursor solutions, and their corresponding SEM images in (h) and (i), respectively.

The morphology, durability, and uniformity of the textile before and after radiative heating is studied by optical microscopy, FT-IR, tensile tests, and XRD, as shown in **Figure 3**. Optical microscope images (Figure 3a and 3b) further illustrate that the textile is not damaged or carbonized after radiative heating. The brown color of the substrate after radiative heating, again, indicates the formation of Pt nanoparticles that absorb visible light, rather than potential damage to the textile (refer to Figure S5 for the optical microscopic images of a pristine substrate before and after radiative heating). The functional groups of these samples were also tested by FT-IR spectroscopy (Figure 3c). The O-H ( $3328.58\text{ cm}^{-1}$ ), C-H ( $2913.96\text{ cm}^{-1}$ ), C=O ( $1712.5\text{ cm}^{-1}$ ), and C-O ( $1241.95\text{ cm}^{-1}$ ,  $1095.39\text{ cm}^{-1}$ ,  $1016.32\text{ cm}^{-1}$ ) remain the same after the radiative heating process, proving negligible carbonization during the radiative heating process.<sup>35</sup> In addition, the mechanical strength of textile samples before and after radiative heating was measured by a Tinius Olsen H5KT tester. As shown in Figure 3d, the strength of the Pt nanoparticle loaded textile (45.9 MPa) is similar to that of the pristine textile (50.7 MPa), indicating the physical structure of the textile is maintained after the ultrafast radiative heating process. We also verified the structure of the textile before and after radiative heating by XRD (Figure S2). In the XRD spectra, the peaks at  $13.64^\circ$ ,  $16.76^\circ$ , and  $22.67^\circ$  belong to cotton, and the  $26.01^\circ$  peak is attributed to polyester,<sup>36</sup> which persist after the radiative heating process. All the above results from microscopy images, FT-IR, tensile tests, and XRD illustrate that the physical and chemical structure of the textile is retained after the ultrafast radiative heating process.

The uniformity of the Pt nanoparticles throughout the textile substrate is verified to understand the directionality of the radiative heating process. As the radiative heating process is applied to only one side of the substrate, the surface area indirectly heated by the radiative source is

investigated by SEM. On the fiber oriented away from the radiative heating source, the Pt nanoparticles are uniformly dispersed with morphologies consistent with nanoparticles directly facing the radiative heating source (Figure 3e). Nanoparticles formed along the middle of the fiber are also similar in size and dispersion from the rest of the textile (Figure 3f, also Figure S6). Therefore, at an active working distance of 0.5 cm, there is little to differentiate the nanoparticles given their location on the textile. These observations indicate that the synthesis is relatively invariable with the orientation of the radiative heating source, so long as the intensity of the radiative source is high enough to prompt thermal decomposition of the precursor materials on the surface of the textiles.



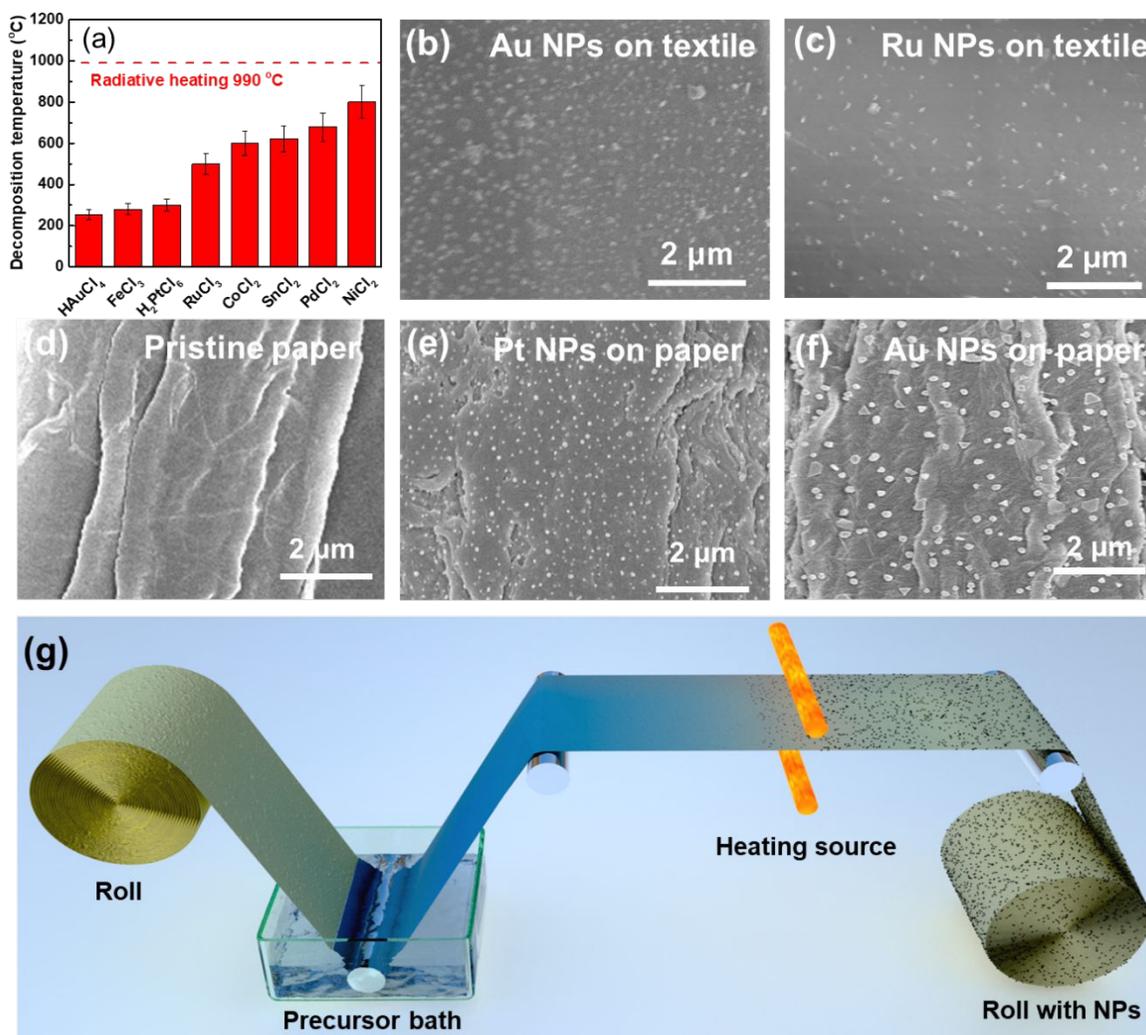
**Figure 3.** Morphological and structural evolution of the textile before and after radiative heating. (a, b) Microscopy images of the textile with precursors before and after radiative heating, respectively, the black color is due to the synthesis of nanoparticles on the textile after radiative heating; (c) FT-IR of the pristine textile, the textile with Pt precursor, and the textile after radiative heating; (d) the mechanical strength of the textile with Pt precursors before and after radiative heating; (e, f) the uniformity of the Pt nanoparticles along the top and middle orientations of the textile (refer to the radiative source).

Given the temperatures (990 °C) achieved during the radiative heating method, this approach can be adapted to form various metal nanoparticles on low-T substrates by thermal decomposition of other salt precursors. As shown in Figures 4b and 4c, Au and Ru nanoparticles can be uniformly dispersed on textile substrates by the same radiative heating process, thereby demonstrating the transferability of this method to other material systems. As with the Pt nanoparticle based textiles, the substrates blanketed with Au and Ru nanoparticles show negligible degradation after radiative heating. However, the precursor salts do show varying decomposition temperatures and affinities to the low-T substrates which is manifested by varying particle sizes and distributions.<sup>37</sup>

The structural integrity of fragile low-T substrates after the rapid 'fly-through' synthesis is one of the most surprising and exciting aspects of this approach and also can be easily extend to other substrates. Feather-weight paper (a typical low-T substrates) used in these experiments are loosely knit to better store the precursor salts without any additives on the surface of the substrate (**Figure 4d**). Using the same radiative heating process, Pt and Au nanoparticles can be uniformly dispersed on the feather-weight paper substrate with a size distribution between 50 - 80 nm and 100 - 240 nm, respectively. (Figure 4e and 4f). The differential particle diameters of the Pt and Au nanoparticles are decided by the decomposed temperature and the affinity of the metals. Therefore, this rapid radiative heating method is an appealing method to synthesize uniform nanoparticles on the porous and flexible low-T substrates.

The ultrafast, roll-to-roll manufacturing of the rapid radiative heating process for low-T substrates is envisioned using the schematic in Figure 4g. In the roll-to-roll process, a low-T substrate is immersed in a precursor bath and then passes by the radiative heating source for rapid synthesis of metal nanoparticles. In particular, this approach is made possible by the flexibility of the low-T substrates and the rapid, non-contacting radiative heating method. Therefore, the

synthesis of nanoparticles on the low-T substrates through the rapid and non-contact radiative heating method is compatible with existing industrial protocols, which holds great promise towards ultrafast, roll-to-roll nanomanufacturing of various supported nanoparticles.



**Figure 4.** The universality of the radiative heating on flexible substrates. (a) The decomposition temperature for different precursor salts, which usually can be decomposed under the radiative heating temperature. The microstructure of pristine paper (d) as well as paper loaded with Pt (e) and Au (f) nanoparticles. (g) Schematic of roll-to-toll manufacturing of nanoparticles by the radiative heating method on low-T substrates.

## Conclusions

For the first time, we report a “fly-through” method to synthesize uniform nanoparticles on temperature-sensitive substrates (e.g. textiles and paper) by the rapid high-temperature radiative heating. The high temperatures achieved from the heating source lead to the thermal decomposition of the precursor salts and formation of metal nanoparticles on the surface of the substrates. The ultrafast radiative heating process ensures the low-T substrates (textiles and papers) remain intact after nanoparticle formation. The non-contact “fly-through” method offers a robust and efficient way to synthesize supported nanoparticles on cheap, flexible, and temperature-sensitive substrates. This continuous particle production method is also deemed compatible with conventional roll-to-roll processes for scalable nanomanufacturing.

## Experimental Section

### *Preparation of the metal precursor loaded textile and paper*

Prepare 0.05 mol L<sup>-1</sup> H<sub>2</sub>PtCl<sub>6</sub> solution, 0.05 mol L<sup>-1</sup> H<sub>2</sub>AuCl<sub>6</sub> solution, and 0.05 mol L<sup>-1</sup> RuCl<sub>3</sub> solution (ethanol was used as the solvent) as the precursor of Pt, Au, and Ru nanoparticles, respectively. The textile in the experiments, composed of cotton and polyester, is cut into 5 cm × 1 cm strips. The textile strips are immersed into the precursor solution and soaked for 20 min, then dried for 1 hour at room temperature. The as-prepared precursor loaded textile was used without further modification for the rapid radiative heating experiments. The feather-weight papers were tested as another low-T substrates, using the same method as the textile specimens.

### *Preparation of the radiative heating source*

The radiative heating source consists of PAN-based carbon nanofibers (CNF), which are prepared by electrospinning. The 8 wt % Polyacrylonitrile (PAN) in dimethylformamide (DMF)

solvent is electrospun at a voltage of 10 kV, a spinning distance of 15 cm, an extrusion rate of 1 mL h<sup>-1</sup>, and a collector rotation speed of 80 rpm. Then, the electrospun fibers are stabilized at 240 °C for 6 h in air and carbonized at 900 °C for 2 hours in argon. The resultant carbon nanofiber mat is cut into 2 cm × 0.5 cm strips and attached to copper electrodes with the silver paste. The carbon nanofibers are capable of bearing a high joule heating load with a local temperature of 3000 K. A Keithley 2425 Source Meter serves as the external electrical power source.

#### *Rapid radiative heating process*

The rapid radiative heating process was conducted in an argon filled glovebox. When the radiative heating source reaches 2000 K, the precursor loaded textile is continuously pulled across the high temperature heating zone at a distance of 0.5 cm with a speed of 0.5 cm s<sup>-1</sup>. The power of the external power source, the distance between the heating source and textile samples, and the velocity of the textile samples across the heating zone can be tuned to vary the working temperature and the reaction time, through which the metal nanoparticles loaded textile can be fabricated evenly. The Au and Ru NPs loaded textiles are prepared by the same method as well as the porous Pt and Au NPs loaded paper samples.

#### *Characterization*

The morphology of the prepared samples was observed by a Tescan XEIA FEG SEM at 5 kV. Mechanical tensile properties were measured on a Tinius Olsen H5KT tester, and FT-IR spectrum was carried out on a Thermo Nicolet NEXUS 670 FT-IR. The X-ray diffraction (XRD) was tested by a D8 Advanced system (Bruker AXS, WI, USA) using a Cu K $\alpha$  radiation source at 40 kV and 40 mA. A thermoelectric couple was used to test the temperature at a certain distance from the heating source, which is the operating heating temperature of the rapid radiative heating method.

The temperature of the heating source was tested by collecting the emitted light (Ocean Optics spectrometer) and fitting the emitted light spectrum to a gray body radiation curve.

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