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DOI

[10.1117/12.2592308](https://doi.org/10.1117/12.2592308)

Publication date

2021

Document Version

Final published version

Published in

Optical Measurement Systems for Industrial Inspection XII

Citation (APA)

Wang, X., Benedictus, R., & Groves, R. M. (2021). Light scattering by gold nanoparticles cured in optical adhesive at optical fibre interfaces. In P. Lehmann, W. Osten, & A. A. Goncalves (Eds.), *Optical Measurement Systems for Industrial Inspection XII* (Vol. 11782). [1178211] (Proceedings of SPIE - The International Society for Optical Engineering; Vol. 11782). SPIE. <https://doi.org/10.1117/12.2592308>

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Xiang Wang, Rinze Benedictus, Roger M. Groves, "Light scattering by gold nanoparticles cured in optical adhesive at optical fibre interfaces," Proc. SPIE 11782, Optical Measurement Systems for Industrial Inspection XII, 1178212 (20 June 2021); doi: 10.1117/12.2592308

SPIE.

Event: SPIE Optical Metrology, 2021, Online Only

Light scattering by gold nanoparticles cured in optical adhesive at optical fibre interfaces

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ABSTRACT

This study forms a part of the research in using nanoparticles (NPs) to increase the intensity of light scattering signal in the optical fibres. Increasing the intensity of the backscattered light signal in the optical fibres shows the potential to increase the signal-to-noise ratio in order to improve the sensitivity of the backscatter reflectometry. Doping NPs into the optical fibres can greatly increase the scattered light. However, it is not easy to manufacture NP-doped optical fibres to test different designs. To overcome this problem, in our former work we used the method of dropping refractive index matching liquid containing gold NPs at the optical fibres end tips to investigate the intensity change of the scattered light from the interfaces. In this paper, some new initial experimental results for the scattered light between the optical fibre end tips are shown. Gold NPs have been mixed into the optical adhesive (Norland) and is then dropped and cured at the optical fibre end tips. A backscatter reflectometer (LUNA ODiSI-B) was used in the experiment to measure the intensity of scattered light distribution between the optical fibre end tips. We investigated 4 cases of light scattering between the optical fibre end tips: (i) the backscattered light intensity distribution in the case of the air gap between the optical fibre end tips; (ii) the backscattered light intensity distribution with optical adhesive between the optical fibre end tips; (iii) the backscattered light intensity distribution with optical adhesive containing gold NPs (gold nanopowder (<100 nm), Sigma Aldrich) between the optical fibre end tips before curing process and (iv) the backscattered light intensity distribution with optical adhesive containing gold NPs between the optical fibre end tips after the curing process. Our initial findings are that the scattered light by gold NPs at the optical fibre interfaces can be detected by the backscatter reflectometer. By obtaining the differential signal between the distributed light scattering by cured optical adhesive containing gold NPs and only optical adhesive between the optical fibre end tips, the light scattered by the gold NPs has been determined.

Keywords: Light scattering, gold nanoparticle, optical adhesive, curing process

1. INTRODUCTION

Strain based Structural Health Monitoring (SHM) has been widely used in the aerospace engineering.^{1,2} Backscatter reflectometry is one of the techniques to detect distributed strain information along the optical fibre.³ Signal-to-noise ratio is a key parameter for backscatter reflectometry, as it restricts the minimal strain signal can be detected. The signal-to-noise ratio can be improved by increasing the backscattered light signals in the optical fibre in the backscatter reflectometer.⁴ Doping nanoparticles (NPs) into the optical fibre⁵ can increase the scattered light dramatically.⁶⁻⁸ The challenge is to optimise the scattered light by the NPs in the optical fibre. However, it is not easy to manufacture NP-doped optical fibres to test different designs in the an optical laboratory. To overcome this problem, we used the method of dropping refractive index matching liquid containing gold NPs at the optical fibres end tips to investigate the intensity change of the scattered light from the interfaces in our previous work⁹ and intend to build on this relationship in the light scattering between the optical fibre end tips and in the optical fibre itself in order to recover the scattered light signal in the optical fibre containing NPs. Although refractive index liquid can have a good refractive index match with the optical fibre, traditional

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optical fibre sensors used for SHM are solid sensors rather than liquid sensors. In this work, nanoparticles will be introduced into the optical adhesive first. After the curing process, the optical adhesive containing gold NPs can be a solid part connecting the optical fibre end tips which is prepared for the strain detection in the future.

This paper includes six sections. Section 1 is the introduction. The methods and results of preparing optical adhesive containing gold NPs are shown in Section 2. The experimental setup and the experimental process are described in Section 3. Section 4 and Section 5 show the results and discussion respectively. Section 6 is the conclusion.

2. PREPARING GOLD NANOPARTICLE ADHESIVE

2.1 Experiment materials and equipment

The experiment materials and equipment mainly include gold NPs (nanopowder, <100 nm particle size, Sigma-Aldrich), optical adhesive (146H, Norland Products) and an ultrasonic processor (750 Watt Ultrasonic Processor, Cole-Parmer).

2.2 Method and results

0.010 g gold NPs was poured into a small glass bottle containing 1 ml optical adhesive. The volume ratio of gold to the optical adhesive is about $5.18 \times 10^{-2} \%$. Then, the gold NPs were dispersed in the optical adhesive by the ultrasonic wave generated by the ultrasonic processor. To avoid overheating of the optical adhesive, the ultrasonic processor paused for 55 s after it ran 5 s. The total running time of the ultrasonic processor was 240 s. The final dispersed gold NPs suspension in the optical adhesive is shown in Figure 3. The red colour shows the scattering and absorption characteristics of the gold NPs in the optical adhesive.

Figure 1: Gold nanoparticles dispersed in the optical adhesive in a glass bottle.

3. DROP GOLD NANOPARTICLE ADHESIVE AT FIBRE INTERFACES

3.1 Experimental setup

A schematic of the experimental setup is shown in Figure 2. A LUNA ODiSi-B was used to extract the backscattered light along the optical fibre, especially to obtain the backscattered light between the optical fibre end tips. One end tip of a single mode optical fibre was connected to the LUNA system. The other end tip of it was fixed on one holder on the upper side of a 3-axis stage (MAX373D, Thorlabs). One end tip of another single mode optical fibre was also fixed on one holder on the 3-axis stage but the position is at the lower side. The other end tip of it was connected to a photodetector (PM20, Thorlabs) to adjust the alignment of the optical fibres in the stage. In the alignment process, a light source (FESL-1550-20-BTF, Frankfurt Laser Company) was used instead

of the LUNA ODiSi-B (not shown in Figure 2). When the alignment process was completed, the photodetector was removed. A syringe controlled by the PreciFluid volumetric dispenser (PreciFluid) and an ultraviolet lamp (PH135 SX Super Xenon, LABINO AB) were positioned close to the optical fibre interfaces. The precifluid volumetric dispenser was used to drop optical adhesive containing gold NPs which has been prepared by the above preparation steps. The ultraviolet lamp was used to cure the optical adhesive at the optical fibre end tips.

Figure 2: Schematic of the experimental setup.

Figure 3: A photo of the fibre interface part of the experimental setup.

Figure 3 shows a photo of fibre interface part of the experimental setup.

3.2 Experimental process

There are 4 experimental processes. The first process is detecting the backscattered light around the two optical end tips with LUNA system, which corresponds to case (i). In case (i), there is only air between the two optical fibre end tips. An example is shown in Figure 3.

The second process is dropping optical adhesive directly between the optical end tips, which corresponds to case (ii). Because the refractive index of the optical adhesive is 1.46 which matches the refractive index of the optical fibre, the backscattered light will decrease dramatically. In this case, the previous FC/APC (ferrule connector/angled physical contact) lower optical fibre end tip was replaced by a FC/PC (ferrule connector/physical contact) connector to increase the backscattered light by the lower optical fibre end tip. Therefore, the reflection from the lower optical fibre can be detected by the LUNA system. The detected signal will be used as the background signal.

The third and the fourth processes are to drop optical adhesive containing gold NPs to the optical interfaces and then use the ultraviolet lamp to cure the adhesive. The backscattered light is detected by the LUNA system. When the distributed backscattered light signal in the case of the optical adhesive containing gold NPs is detected, the detected signal is found by subtracting the background light signal between the optical interfaces.

4. RESULTS

Figure 4a shows the backscattered light signals around the optical end tips detected by the LUNA system. Only air is between the two optical end tips. The intensity values shown in Figure 4a are the original data from LUNA system. The reflected signal from the upper optical fibre end tip is about 165 dB and the reflected signal from the lower optical fibre end tip is about 150 dB. Note: for commercial optical fibre, the backscattered light signal is about -100 dB/mm.¹⁰ Therefore, the values read from the LUNA system directly are not the absolute value of the signals. The values can only be used to show the relative reflected light change.

Figure 4b shows the backscattered light signals after dropping optical adhesive between the optical end tips. As shown in Figure 4b, the reflection from the upper optical fibre end tip reduced by about 27 dB because the refractive index of the optical adhesive matches the refractive index of the optical fibre. Because the low reflection from the lower optical fibre end tip, the reflected light from the lower optical fibre end tip is not easy to be distinguished from the baseline. Therefore, a FC/PC connector was used to replace the lower optical fibre end tip, which was not shown in Figures 2 and 3. In Figure 4b, the high reflection at the lower optical fibre end tip shows the position of the lower optical fibre end tip.

Figure 4c shows the result of backscattered light signals after curing process in blue lines, which is the averaged result from 6 data. Figure 4d shows the intensity difference between the cured optical adhesive containing gold NPs and the only optical adhesive between the optical fibre end tips.

5. DISCUSSION

The intensity of backscattered light by the NPs was obtained in Figure 4d. The highest signals are near the upper optical fibre end tip and decrease exponentially along the optical fibre end tips because of the light extinction by the NPs and only a small amount of the light was recoupled backward into the optical fibre. The 10 dB loss from the baseline after the lower optical fibre end tip also indicates the high light power loss.

Figure 4d was obtained by comparing the results between the cured optical adhesive containing gold NPs and only optical adhesive between the optical fibre end tips. The idea situation is to use the results of the cured optical adhesive containing gold NPs subtract the only cured optical adhesive between the optical fibre end tips, but it is difficult to implement.

Figure 4c shows the result of backscattered light signals after the curing process. It can be seen that the stability of the signal between the optical fibre is similar to that in the optical fibre, but the intensity of the backscattered light signal increased greatly. The LUNA ODiSI-B cannot detect the strain signal between the optical fibre end tips because it is over a very short distance (about 1 mm) and there are two peaks. Therefore, the strain stability test has not been completed successfully with this structure using LUNA system.

Figure 4: Detected backscattered light around the optical fibre end tips by LUNA ODISI-B. (a) Air gap between the optical fibre end tips. (b) Optical adhesive between the optical fibre end tips. (c) Cured optical adhesive containing gold nanoparticles between the optical fibre end tips in blue lines. (d) The intensity differential signals between cured optical adhesive containing gold nanoparticles and only optical adhesive.

The baseline fluctuation from the LUNA ODISI-B causes a difficulty in comparing the results between different tests. It can be seen that the baseline in Figure 4a is about 10 dB larger than the baselines in Figure 4b to 4c. Figure 4b to 4c were obtained in a short time in a sequential process, so the baseline differences are small, but there was a long time gap between obtaining Figure 4a and Figure 4b. It is a drawback of this experiment, so it is a challenge to use LUNA ODISI-B to accurately extract scattered light by the NPs.

6. CONCLUSION

Gold nanopowder has been mixed into the optical adhesive by the ultrasonic processor successfully. By curing, the optical adhesive containing gold NPs formed a solid part connecting the optical fibre end tips which is prepared for the strain detection in the future. The spectra of the light distribution between the optical fibre interfaces can be obtained by LUNA ODISI-B. By obtaining differential signal, the light scattered by the NPs in the optical adhesive was obtained in the experiments. The increased signal by the NPs was about 37 dB in this experiment.

ACKNOWLEDGMENTS

The authors gratefully acknowledge financial support from China Scholarship Council (No.201806020197).

REFERENCES

- [1] Di Sante, R., “Fibre optic sensors for Structural Health Monitoring of aircraft composite structures: Recent advances and applications,” *Sensors* **15**(8), 18666–18713 (2015).
- [2] Kesavan, A., John, S., and Herszberg, I., “Strain-based Structural Health Monitoring of complex composite structures,” *Structural Health Monitoring* **7**(3), 203–213 (2008).
- [3] Froggatt, M. and Moore, J., “High-spatial-resolution distributed strain measurement in optical fiber with Rayleigh scatter,” *Applied Optics* **37**(10), 1735–1740 (1998).
- [4] Loranger, S., Gagné, M., Lambin-Jezzi, V., and Kashyap, R., “Rayleigh scatter based order of magnitude increase in distributed temperature and strain sensing by simple UV exposure of optical fibre,” *Scientific Reports* **5**(1), 1–7 (2015).
- [5] Blanc, W., Mauroy, V., Nguyen, L., Shivakiran Bhaktha, B., Sebbah, P., Pal, B. P., and Dussardier, B., “Fabrication of rare earth-doped transparent glass ceramic optical fibers by modified chemical vapor deposition,” *Journal of the American Ceramic Society* **94**(8), 2315–2318 (2011).
- [6] Beisenova, A., Issatayeva, A., Iordachita, I., Blanc, W., Molardi, C., and Tosi, D., “Distributed fiber optics 3D shape sensing by means of high scattering NP-doped fibers simultaneous spatial multiplexing,” *Optics Express* **27**(16), 22074–22087 (2019).
- [7] Ayupova, T., Shaimerdenova, M., Korganbayev, S., Sypabekova, M., Bekmurzayeva, A., Blanc, W., Sales, S., Guo, T., Molardi, C., and Tosi, D., “Fiber optic refractive index distributed multi-sensors by scattering-level multiplexing with MgO nanoparticle-doped fibers,” *IEEE Sensors Journal* **20**(5), 2504–2510 (2020).
- [8] Tosi, D., Molardi, C., and Blanc, W., “Rayleigh scattering characterization of a low-loss MgO-based nanoparticle-doped optical fiber for distributed sensing,” *Optics & Laser Technology* **133**, 106523 (2021).
- [9] Wang, X., Benedictus, R., and Groves, R. M., “Light scattering and rheological effects in an optical fibre coupled nanoparticle suspension,” in [*Optical Sensing and Detection VI*], **11354**, 113540V, International Society for Optics and Photonics (2020).
- [10] Lu, P., Mihailov, S. J., Coulas, D., Ding, H., and Bao, X., “Low-loss random fiber gratings made with an fs-IR laser for distributed fiber sensing,” *Journal of Lightwave Technology* **37**(18), 4697–4702 (2019).