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Distributed hydrogen sensing using in-fiber Rayleigh scattering

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This letter reports a fully distributed hydrogen sensing technique using Rayleigh backscattering in palladium (Pd) and copper (Cu) coated optical fiber. The local in-fiber strain changes due to Pd hydrogen absorptions are interrogated spatially resolved optical frequency domain reflectrometry measurements of the Rayleigh signals. Electrical power is used to induce heating in the Pd coating, which accelerates both the hydrogen response and the sensor recycling. This technique promises an inexpensive and truly distributed fiber solution for continuous hydrogen leak detection with centimeter spatial resolution at room and low temperatures. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4712592]

Hydrogen has wide applications in chemical and energy industry. In recent years, it has also become one of the major candidates of clean energy sources. However, hydrogen is an extremely reactive gas, which can be ignited in air from 4% to 75%. The ignition energy is as small as 0.02 mJ, and the flame velocity is almost ten times larger than that of natural gas. Therefore, fast and accurate detection of hydrogen gas leak in both room and cryogenic temperatures is critically important for the safety of hydrogen production, transportation, and utilization. A majority of hydrogen sensors developed in the last 20 years have been based on the fast and selective absorption of hydrogen with palladium (Pd). Due to the explosion-proof nature of optical fiber, many Pd-coated optical fiber sensors have been demonstrated for single-point hydrogen sensing, including Pd-coated mirror,[1] side-polished and tapered fiber sensor,[2,3] Fabry-Perot interferometer (FPI),[4] and fiber Bragg gratings (FBGs).[5–8] To enhance the hydrogen absorption rate of Pd in low temperature, different in-fiber heating techniques have been developed using long period grating (LPG),[5] double-clad fiber (DCF),[6] and high attenuation fiber (HAF).[7,8]

A more challenging issue is to develop distributed sensing technology, so sophisticated hydrogen system can be completely monitored using a single interrogation unit. Multipoint quasi-distributed hydrogen sensing has been reported with FPI sensors multiplexed in parallel with fan-out fiber coupler[4] and FBG sensors multiplexed in serial on a single fiber,[5–8] both of which provide excellent centimeter spatial resolution with their compact sizes. However, the distributed sensing capabilities of FPI and FBG point sensors are fundamentally limited by their multiplexing density and the consequent high manufacturing cost. A more efficient approach is to use the optical fiber itself as a continuous hydrogen sensing device. Sumida *et al.*[9] reported 1-m spatial sensing resolution in a Pd-coated fiber evanescent field sensor design using optical time domain reflectometry (OTDR). More recently, Wang *et al.*[10] enhanced the sensing resolution to 10-cm using acoustically generated traveling LPG in Pd-coated fiber.

In this letter, we reported 1-cm-resolution distributed hydrogen sensing based on optical frequency domain reflectometry (OFDR) measurements of Rayleigh scattering in Pd/Cu coated optical fibers. This technology has recently been applied to provide distributed sensing solution for temperature and axial strain measurements.[11–13] Herein, Pd/Cu coated single-mode fibers are used to extend its application for distributed hydrogen sensing. By measuring the in-fiber Rayleigh spectral shift, local axial strain induced by Pd-hydrogen absorption is interrogated in 1-cm resolution over a piece of fiber under test (FUT). At room temperature, hydrogen sensing operations are performed for hydrogen concentration from 1% to 10% and sensing time from 1 to 20 min. It is also shown that the hydrogen sensitivity is significantly enhanced with distributed on-fiber heating, which improves the low temperature sensing performances. By using the optical fiber itself as a fully distributed sensing element, the technique demonstrated in this letter completely eliminates the need of using individual FBG and FPI point sensors and cost associated with the manufacturing process. Hereby, it provides a practical and inexpensive solution for distributed hydrogen leak detection with a record-breaking distributed spatial resolution comparable to point sensors.

As shown in Fig. 1, proof-of-concept experiments were performed at room temperature (20 °C) using a commercial optical backscatter reflectometer (OBR) from Luna Technologies

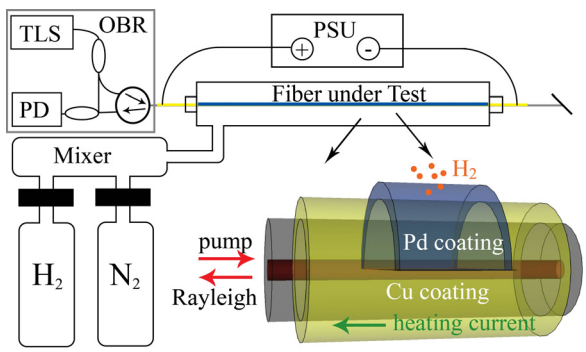

FIG. 1. Schematic of the distributed hydrogen sensing system using electrically heated optical fiber. OBR: optical backscatter reflectometer; TLS: tunable laser source; PD: photodiode; and PSU: power supply.

(OBR 4600) (Ref. 13) connected with a piece of FUT. The single-mode silica FUT was coated with 20-μm thick Cu alloy and further sputter-coated with 1-μm thick palladium on one side of the fiber surface. 2-m long Pd/Cu fiber coatings were fabricated in the laboratory with good uniformity. Longer and distributed coatings are also possible. A 350-mm long Pd/Cu coated FUT is inserted into the gas test chamber and sealed at both ends with fiber ferrules. Gases from a pure nitrogen gas tank and a 10% concentration hydrogen gas tank are pre-mixed and sent into the test chamber at atmosphere pressure (14.7 psi or 1 bar). The hydrogen concentration under test is controlled with two mass flow meters. The FUT can be electrically heated using a regulated current supply.

The OBR unit consists of a tunable laser source continuously sweeping from 1520 to 1610 nm, a fiber interferometer with measurement arm connected to the FUT, and a pair of highly sensitive photodiodes (PDs) with sampling rate of 2 MS/s. The excited Rayleigh scattering and possible discrete reflections in the FUT are reflected back to the OBR and recombined with a small portion of sweeping light in the reference arm, which at the moment of the recombination is at different wavelengths due to the unbalance path in measured and reference arms.

For each Rayleigh scattering location along the FUT, the wavelength difference encodes the location information into a unique beating signal, which is received using the PDs, and spatially interrogated afterwards using fast Fourier transform upon the sweeping wavelength. By cross-correlating with a pre-measured reference, Rayleigh spectral shifts induced by external perturbations along the FUT can also be spatially interrogated. The spatial resolution is determined by the wavelength sweeping range and the size of cross-correlating window.[12,13] A 1-cm long cross-correlating window is chosen in the experiment to obtain ~1-pm spectral resolution, which corresponds to ~0.67 $\mu\varepsilon$ strain resolution for the FUT at 1550-nm wavelength. This choice of window size is a careful balance between the required spatial resolution and the Rayleigh sensitivity needed for reliable detection of hydrogen below 4% threshold.

First, the on-fiber heating performance of the sensor is tested in 100% nitrogen. The distributed Rayleigh spectral shifts measured with OFDR are associated with in-fiber temperature changes. Thermal-optic coefficient of 0.06 °C/pm is used for the 20-μm thick copper coated fiber,[13] which is approximately half of the value for plastic coated SMF-28 fiber at room temperature. The additional thermal expansion effect from 1-μm thick one-side Pd coating is neglected. Both the heating profile along the fiber and the heating efficiency is illuminated in Fig. 2 for different heating current and electrical power densities. Uniform, linear and efficient electrical on-fiber heating is observed along the FUT at smaller current. The fiber can be heated up to 146 ± 5 °C using 5.24 mW/mm electrical power.

To test the distributed hydrogen sensing capability, two portions of the FUT at 2290–2330 mm and at 2460–2520 mm, respectively, are exposed to 4% explosive-threshold concentration hydrogen for 2-min long with and without the 5.34 mW/mm on-fiber heating (e.g., ~140 °C on-fiber temperature). The results are shown in Fig. 3. Immediately after the hydrogen exposure and shut-down of the on-fiber heating, the

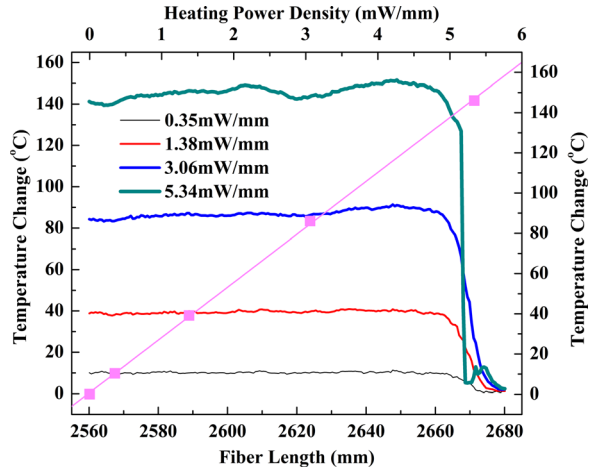

FIG. 2. The temperature change along the fiber with different heating power densities, measured by the in-fiber Rayleigh scattering.

distributed Rayleigh spectral shifts are measured. The measurement results are then computed with pre-measured reference to calculate in-fiber strain changes induced by the Pd hydrogen absorption. The heated sensor response is ~3 times larger than unheated response. And 1-cm spatial resolutions are obtained for both portions on the FUT. The uneven sensor response could come from the un-uniform coating profile of the Pd coating.

The hydrogen sensor responses are also studied systematically for different hydrogen exposure times and different hydrogen concentrations both with and without the 5.34 mW/mm on-fiber electrical heating. The results are detailed in Fig. 4. Fig. 4(a) shows that 10% concentration hydrogen can be readily detected within exposure time as short as 1-min. And for an exposure time of 2-min, hydrogen concentration as small as 1% can be readily detected with the chosen Rayleigh sensitivity, as shown in Fig. 4(b). In both cases, sensor responses are significantly enhanced with on-fiber heating, which eliminates possible detection errors, and more importantly makes this technology promising for low temperature sensing environment.

In summary, we have demonstrated the effective operation of an all-fiber hydrogen sensing system for fully distributed hydrogen leak detection. This technique employs

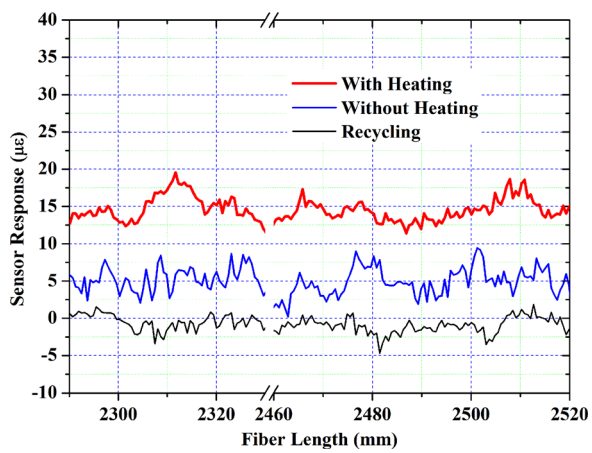

FIG. 3. The sensor strain response to hydrogen exposure at different positions on the same fiber.

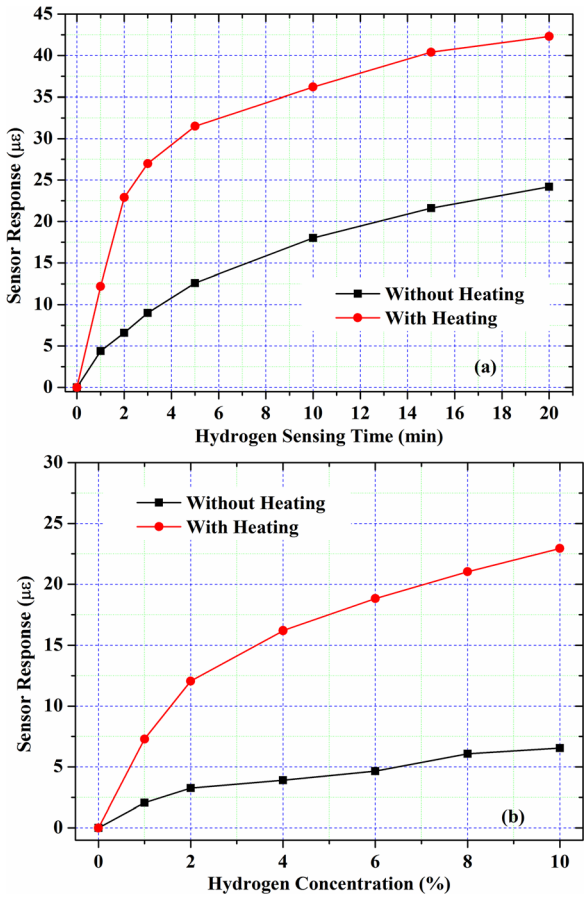

FIG. 4. The sensor response with and without electrical heating to (a) hydrogen exposure time from 0 to 20 min for fixed 10% hydrogen concentration; (b) hydrogen concentrations from 1% to 10% for fixed 2-min exposure time.

wavelength-swept Rayleigh backscattering interferometry in OFDR fashion to obtain 1-cm spatial resolution for hydrogen sensing continuously along a single piece of Pd/Cu coated fiber. The sensor response is enhanced with uniform and efficient electrical on-fiber heating, which makes the sensor feasible for low temperature operation. Further improvements in sensing performances, including response time, sensitivity, and detection range are possible through optimization of the Pd/Cu coating recipe. By tuning the heating current and the thickness distribution of Cu coating along the fiber, the electrical heating can be applied flexibly on optical fiber. Spark hazard can be minimized with additional isolation and below threshold heating current. It is also possible to obtain better spatial range and resolution using OFDR components with faster speed and larger range. Temperature cross-sensitivites can be discriminated from the hydrogen strain responses by employing polarization maintained fiber as FUT.[14] Furthermore, using multimode fibers and high attenuation fibers, optical on-fiber heating[5–8] can be employed instead of electrical heating to completely eliminate the spark hazard, but the sensing length is limited to 100–200 mm long and uniform heating profile cannot be guaranteed. In our opinion, this paper demonstrates a fully distributed all-fiber hydrogen sensing solution with good prospects for practical applications.

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