



# Kinetic control of nanocrack formation in a palladium thin film on an elastomeric substrate for hydrogen gas sensing in air



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## ABSTRACT

We report the effects of various tensile velocities on the nanocrack formation in a Pd thin film on an elastomeric polydimethylsiloxane substrate and its H<sub>2</sub> sensing properties. A tunable nanocrack along the *x* and *y* axes was created by mechanical stretching/compression cycles with varying tensile velocities. From the microstructural analyses, we found that the tensile velocity has a significant effect on the crack density but little effect on the average crack width. The Pd nanogap sensor prepared under a high tensile velocity showed a high performance with a low detection limit of 500 ppm of H<sub>2</sub> in air. Our results indicate that the higher crack density with the narrow nanocrack width (55–100 nm) propagated over the entire film provides the enhanced H<sub>2</sub> sensing properties in air.

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## 1. Introduction

In order to generate structures at the nanoscale, advanced nanopatterning and nanofabrication technologies typically make use of templates in the fields of soft lithography, nanoimprint lithography, and hot embossing. However, there are also lithography-free methods that can create periodic patterns without the use of templates. Wrinkling and cracking are two examples of periodic patterns that occur in stiff films and on elastomeric substrates. These patterns have generated increasing interests for a wide range of practical applications, including photonics [1–4], stretchable electronics [5–9], microfabrication [1,10–16], and flexible sensors [17–22]. The first wrinkle pattern was reported on a 50-μm-thick Au film thermally deposited on a polydimethylsiloxane (PDMS) substrate, where the thermal expansion mismatch between the metal film and the PDMS substrate, caused by cooling the sample surface from 110 °C to room temperature (RT), was used to cause buckling via compressive stresses [10]. Many studies have developed fundamental theories and a variety of experiments aimed at developing multifunctional micro/nanoscale patterns induced by the internal compressive stress [9,23–27]. In contrast, crack patterns are typically formed using the tensile stress. Some studies on crack pattern formation have reported that the

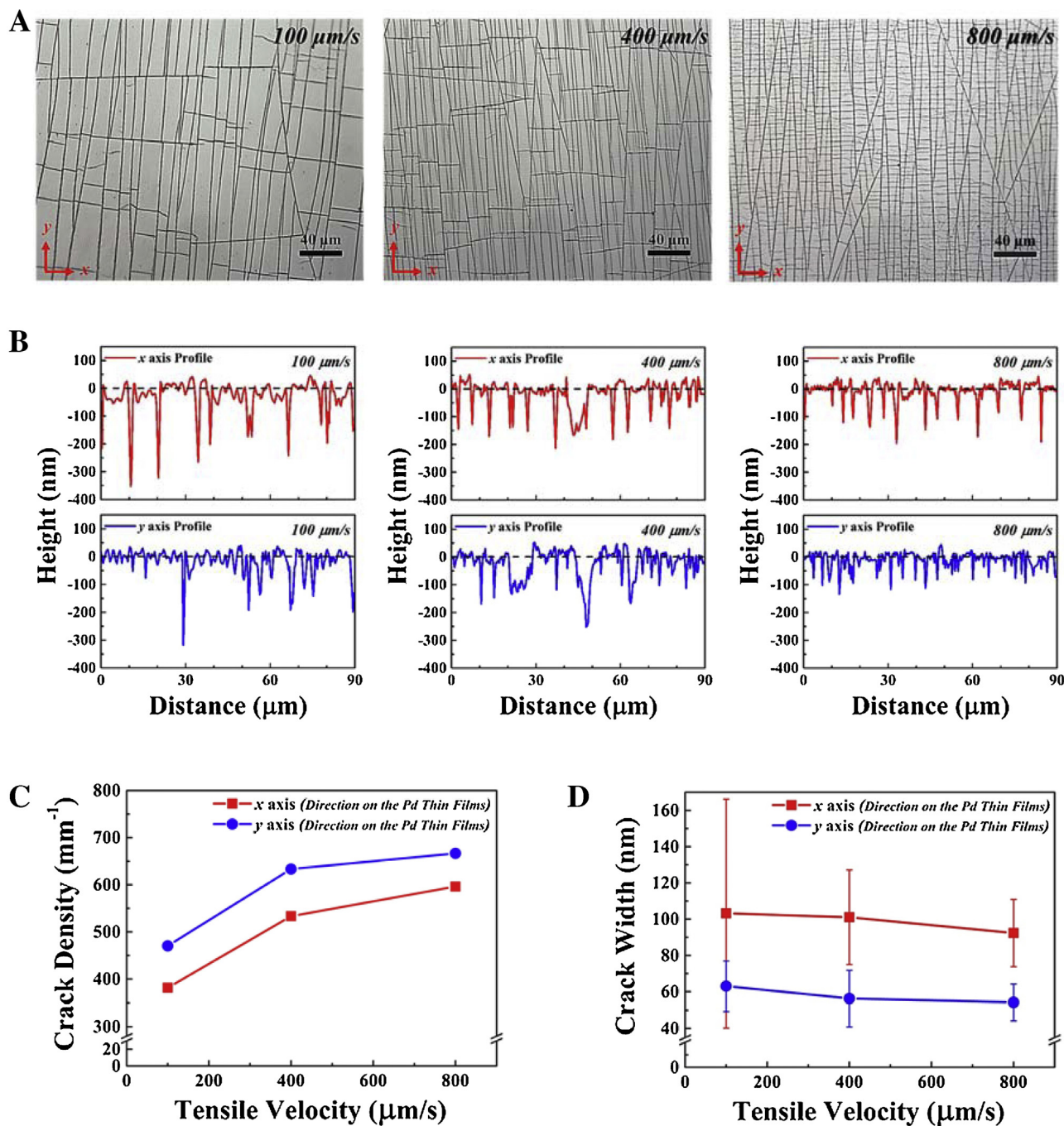
crack pattern can be modulated by altering the mechanical stress applied to the metal/polymer bilayers at ambient temperature. The tensile stress can create an array of nanocracks within the stiff film that are perpendicular to the applied stress because of the differences between the mechanical properties of the metal thin film and those of the elastomeric substrate [28].

In our earlier work, we investigated a nanogap-based sensor in a Pd metal film on a PDMS substrate by applying tensile stress for the first time [29]. Subsequently, qualitative methods have been suggested to develop the nanogap-based sensors using both a highly mobile thin film on an elastomeric substrate by stretching [30–32] and a cracked Pd film on an elastomeric substrate by repeat of H<sub>2</sub> treatment for different H<sub>2</sub> concentrations [33,34] for controlling the crack formation for H<sub>2</sub> sensors. The performance of such sensors was tested in inert N<sub>2</sub> environments. The sensors exhibited superior on–off behavior with a wide range of detection H<sub>2</sub> (10–40000 ppm) in N<sub>2</sub> [29–34]. The studies also reveal that the reduction in the crack width is a significant factor for the detection of a low H<sub>2</sub> concentration under N<sub>2</sub> [29–34]. However, many efforts have not been made to create the nanocracks and control its width or density via a quantitative method for nanogap-based sensors used for H<sub>2</sub> sensor in air atmosphere for practical applications.

In this study, we report the effects of various tensile velocities on the nanocrack formation in a Pd thin film on an elastomeric PDMS substrate and its sensing properties in the air. We demonstrate that intentional mechanical stretching/compression cycles provide tunable nanocrack formation along the *x* and *y* axes which can be controlled due to tensile velocity.

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**Fig. 1.** (A) Perspective-view optical microscopy of nanocracks generated in a Pd thin film on PDMS after applying a tensile stress as a function of the tensile velocity, (B) crack depth distribution along the x and y axes obtained from the atomic force microscopy (AFM) maps, (C) crack density calculated from the AFM depth profiles at various tensile velocities, and (D) average crack width distribution along the x and y axes estimated from an SEM images.

## 2. Experiment

### 2.1. Fabrication of samples

A thin film of PDMS cured at RT for 16 h and then at 75 °C for 3 h was used as an elastomeric substrate (1.0 cm (W)  $\times$  2.7 cm (L), 0.6–0.75 mm thick), and a Pd film was deposited on top of the PDMS substrate via ultrahigh-vacuum (UHV) DC magnetron sputtering in an Ar atmosphere. The Pd film was 1.0 cm (W)  $\times$  1.5 cm (L) in area with a thickness of 10 nm. The deposition process was carried out below  $2.4 \times 10^{-3}$  Torr in Ar at a flow rate of 34 sccm in a vacuum chamber with a base pressure of  $4.7 \times 10^{-8}$  Torr. The purity of the Pd

target was 3 N. The deposition rate of the Pd film at RT was  $\sim 3.7$  Å/s at 20 W.

### 2.2. Nanocrack formation

The mechanical properties of the Pd thin films on PDMS were measured using a microtensile tester (Linkam, TST350) with a force of 10–14 N at constant RT tensile velocities of 100, 400, and 800  $\mu\text{m/s}$ . The samples were strained by 100% over 20 cycles (loading/unloading), which was defined as  $(L - L_0)/L_0 \times 100$  (%), where  $L_0$  and  $L$  are the initial length and the final length with an applied ten-

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