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Report Title

Nanometer-Size Magnetic Devices

ABSTRACT

This research is to understand the relationship between the local magnetic domain changes and the magnetization reversal behaviors of nanometer-sized magnetic features and to develop improved methods for understanding and characterizing the magnetic properties of nanometer-sized materials. We have developed a few advanced cantilevers for magnetic characterization of small magnetic objects. We have studied magnetic layers in magnetic sensors.

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(a) Papers published in peer-reviewed journals (N/A for none)

1. L. Gao, L. Yue, T. Yokota, R. Skomski, S. H. Liou, H. Takahoshi, H. Saito, and S. Ishio, "Focused Ion Beam Milled CoPt Magnetic Force Microscopy Tips for High Resolution Domain Images", IEEE Trans. on Magnetics, 40, 2194 (2004).

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(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)

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1. L. Gao, L. Yue, T. Yokota, R. Skomski, S. H. Liou, H. Takahoshi, H. Saito, and S. Ishio, "Focused ion beam milled CoPt magnetic force microscopy tips for high resolution domain images", Presented at 9th Joint MMM-Intermag Conference, Anaheim, California, January 5-9, 2004.

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 L. Yuan, L. Gao, R. Sabirianov, S. H. Liou, M. D. Chabot, D. H. Min, J. Moreland and Bao Shan Han, "Microcantilever Torque Magnetometry Study of Patterned Magnetic Film", Presented at IEEE International Magnetics Conference, San Diego California, May 8-12, 2006.

14. L. Yuan, Y. S. Lin, Dexin Wang and S. H. Liou, "Tuning Magnetic Microstucture of Reference Layer in Magnetic Tunning Junctions", Presented at 10th Joint MMM-Intermag Conference, Baltimore, Maryland, January 7-11, 2007.

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18. S. H. Liou, Rui Zhang, Stephen E. Russek, L. Yuan, Sean T. Halloran, and David P. Pappas, "Dependence of noise in magnetic tunnel junction sensors on annealing field and temperature" Presented at 52nd Annual Conference on Magnetism and Magnetic Materials, Tampa, Florida, November 5-9, 2007.

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20. S. H. Liou, Rui Zhang, Stephen E. Russek, L. Yuan, Sean T. Halloran, and David P. Pappas "Noise study of magnetic tunnel junction sensors with magnetic flux concentrators", Partners in Environmental Technology Technical Symposium & Workshop December 4-6, 2007, at the Marriott Wardman Park Hotel in Washington, D.C.

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Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

S. H. Liou, Rui Zhang, Stephen E. Russek, L. Yuan, Sean T. Halloran, and David P. Pappas, "Dependence of noise in magnetic tunnel junction sensors on annealing field and temperature" accepted J. Appl. Phys., (2008)
I.-C. Chen, L.-H. Chen, A. I. Gapin, S. Jin, L. Yuan and S.-H. Liou, "Iron-platinum coated carbon nanocone probes on tipless cantilevers for high resolution magnetic force imaging", Nanotechnology (Submitted) (2008).
Number of Manuscripts: 2.00

Number of Inventions:

Graduate Students		
NAME	PERCENT SUPPORTED	
Lan Gao	0.25	
Lu Yuan	0.25	
Danqin Feng	0.10	
LeighAnn Nicholl	0.10	
Rui Zhang	0.10	
Yushun Lin	0.10	
FTE Equivalent:	0.90	
Total Number:	6	

Names of Post Doctorates

	Names of Faculty Sunnarted	
FTE Equivalent: Total Number:		
	FERCENT_SUFFORTED	

NAME	PERCENT SUPPORTED	National Academy Member
Sy-Hwang Liou	0.25	No
Renat Sabirianov	0.10	No
FTE Equivalent:	0.35	
Total Number:	2	

Names of Under Graduate students supported

<u>NAME</u>	PERCENT_SUPPORTED	
Wenjin Zhou	0.10	
FTE Equivalent:	0.10	
Total Number:	1	

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The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields: 1.00				
Number of graduating undergradu	uates who achieved a 3.5 GPA to 4.0 (4.0 max scale): 1.00			
Number of graduating undergraduates fun	ded by a DoD funded Center of Excellence grant for Education, Research and Engineering: 0.00			
The number of undergraduates funded by your a	agreement who graduated during this period and intend to work for the Department of Defense 0.00			
The number of undergraduates funded by your agr	eement who graduated during this period and will receive			
scholarships or fellowships for further studies	in science, mathematics, engineering or technology fields: 1.00			
Names of Personnel	receiving masters degrees			
NAME				
Rui Zhang				
Danqin Feng				
Yushun Lin				
Total Number: 3				
Names of personnel receiving PHDs				
NAME				

Sub Contractors (DD882)

Names of other research staff

2

PERCENT_SUPPORTED

NAME

Lan Gao Lu Yuan **Total Number:**

FTE Equivalent:

Total Number:

Inventions (DD882)

The problem studied:

This research is to understand the relationship between the local magnetic domain changes and the magnetization reversal behaviors of nanometer-sized magnetic features and to develop improved methods for understanding and characterizing the magnetic properties of nanometer-sized materials. We have developed a few advanced cantilevers for magnetic characterization of small magnetic objects. We have studied magnetic layers in magnetic sensors.

A few examples as follows:

- (1) Magnetic properties of ultra-thin CoPt films with Au layer inserted
- (2) Magnetic Force Microscopy Study of CoPtCrO Perpendicular Media With Superparamagnetic And Permanent Magnet Tips
- (3) Focused Ion Beam Milled CoPt Magnetic Force Microscopy Tips for High Resolution Domain Images
- (4) Batch Fabricated High-resolution MFM Tips for Both Soft and Hard Magnetic Materials
- (5) Microcantilever Torque Magnetometry Study of Patterned Magnetic Films
- (6) Torsion Oscillator Magnetic Field Sensors
- (7) Imaging Magnetic Noise Sources in Magnetic Recording Heads
- (8) Dependence of Noise in Magnetic Tunnel Jjunction Sensors on Annealing Field and Temperature

Summary of the most important results: (1) Magnetic properties of ultra-thin CoPt films with Au layer inserted

We investigated the reduction in the ordering temperature using a CoPt multilayer with Au layer inserted. It was revealed that the adding of Au is effective for reducing the ordering temperature by about 200 0 C. From the structural analysis, the phase formation of the L1₀ structure of the Au inserted CoPt layer films starts at around 350 ~ 400 °C. At the same temperature, the H_c of these films has drastic changes. It shows clearly that the occurrence of high H_c is related to the phase formation of L1₀ structure of CoPt. The study of the time dependence of H_c reveals that the phase formation of L1₀ structure of CoPt is a slow diffusion process.

Fig. 1 shows H_c at 300 K versus the annealing temperature (T_a) for the CoPt film with or without Au layer inserted. For as-deposited CoPt films, both with and without Au layers, the H_c at 300 K is about 30 Oe and the M-H curves exhibit soft magnetic properties. After annealing, the H_c of pure CoPt film changed slowly. The H_c of the pure CoPt film annealed at 550 °C is about 2 kOe. The H_c of the CoPt (3 nm)/Au (2 nm) multilayer films began to change at 350 °C and drastically increase over 400 °C. The H_c of the CoPt (3 nm)/Au (2 nm) multilayer sample annealed at 350 °C is about the same as that of pure CoPt films annealed at 550 °C. These results indicated clearly the effect of adding Au for the reduction of the processing temperature. Such a remarkable improvement of processing temperature due to the addition of Au can be better understood by the temperature dependence of structure changes. Fig. 2 shows the change in the d-spacing (d₁₁₁) versus T_a. The d-spacing of the CoPt (3 nm)/Au (2 nm) multilayer samples is rapidly decreased around 400 °C. The change in the lattice parameter has the same tendency as that of the H_c. Judging from these results, the phase formation of the L1₀ structure of the CoPt inserted Au layer films starts around 350 ~ 400 °C. And, in the sample with a 2 nm Au layer, a temperature of 400 °C is enough for a high coercivity (above 5 kOe).



Fig. 1 Annealing temperature (T_a) dependence of the in-plane coercive force (H_c) for the different Au layer thickness. (Square is 2nm, open circle is 1 nm, and triangle is 0 nm, respectability.) The annealing time is 5 hous.



Fig. 2 Change in the d-spacing (d_{111}) versus the annealing temperature (T_a) of the film with 2 nm Au layer with various annealing temperatures $(25 - 550^{\circ}C)$.

(2) Magnetic Force Microscopy Study of CoPtCrO Perpendicular Media With Superparamagnetic And Permanent Magnet Tips

Magnetic force microscopy (MFM) has been widely used in the study of magnetic recording media. It requires high resolution MFM tips as well as better understanding of the obtained magnetic images. In this study, we compared the images obtained by superparamagnetic and permanent magnet MFM tips, which allowed us to explain the issues related to the frequency doubling in some domain images of the recording media. As shown in Fig. 3, we compared the magnetic (phase) image of the 200 kfci track in an ac-erased area taken with a permanent magnet and a superparamagnetic tip. Fig. 3a is the image obtained by the permanent magnet MFM tip, which shows a dominant spectral frequency of about 200 kfci. Fig. 3b is the image taken by a superparamagnetic tip, which shows a dominant spectral frequency of about 400 kfci. The frequency doubling was clearly observed by using a superparamagnetic tip. This may explain the observation of frequency doubling by Zhifeng Deng et al. in their phase images of a PMR medium using metal-coated carbon nanotube tips. We show that the magnetic images obtained by different types of MFM tips can provide valuable magnetic information about the sample.

In summary, to study magnetic images, using more than one type of tip may help in the quantitative analysis of MFM data.



Fig. 3 Magnetic (phase) images of the 200 kfci track in an ac-erased area taken with (a) a permanent magnetic tip and (b) a superparamagnetic tip.

(3) Focused Ion Beam Milled CoPt Magnetic Force Microscopy Tips for High Resolution Domain Images

High coercivity CoPt magnetic force microscope tips have been modified by focused ion beam milling to improve the resolution of magnetic domain images. The magnetic materials around the apex have been removed leaving a 30 nm diameter magnetic particle at the tip end (as shown in Fig. 4). Due to the smaller amount of magnetic material, the stray field from this new tip is significantly reduced and the spatial resolution of magnetic domain images is improved. The tip is used to obtain high resolution domain images of a CoCrPt-SiO₂/Ru perpendicular recording medium with linear recording densities from 800 to 1000 kfci. Magnetic patterns of 900 kfci, corresponding to a bit size of 28 nm, are well resolved. From the analysis of the power spectrum of the track profiles for these images, a spatial resolution as good as 11 nm under ambient conditions with a commercial magnetic force microscope is achieved.

The resolution of the MFM can be characterized in real space by its "point response" or in Fourier transform space by its wave-vector (spatial frequency) response. Essentially, the two approaches are equivalent. Fig. 5 shows the domain images of recording tracks with linear recording densities from 800 to 1000 kfci (kilo flux changes per inch). Tracks of 800, 900 and

1000 kfci correspond to bit sizes of 32, 28 and 25 nm, respectively. The MFM image of the 800 kfci track presents well-resolved recording bits. Track densities up to 900 kfci are clearly visible by MFM. The visibility of the 1000 kfci track transitions is much less pronounced.

Fig. 6(a) shows the power spectrum of the 800 kfci track. The peak corresponds to the recorded signal with a wave length of 73 nm (which is double the recording bit size). This value is higher than the calculated value of 64 nm for 800 kfci. This may be due to inaccuracies in the recording process. As shown in Fig. 6(c), the peak corresponds to the recorded signal of a 1000 kfci track with a wavelength of 58 nm and is only resolved by Fourier transform. In Fig. 6(a), a wavelength cutoff of 22 nm is obtained from the intersection of the signal and the noise. This corresponds to an MFM image resolution of 11 nm, which is half the wavelength. For the 900 and 1000 kfci tracks, the resolutions obtained from the wavelength cutoffs are 12 and 11 nm, respectively, as shown in Fig. 6(b) and 6(c). The error of the resolution is about 2 nm, which is due to the inaccuracies of drawing the signal line and the noise line.



Fig. 4. An FIB milled MFM tip shows a magnetic particle with a diameter of 30 nm at the tip end.



Fig. 5 Magnetic domain images of a recording disk with different writing bit density, 800 kfci, 900 kfci and 1000 kfci.



Fig. 6 An FFT analysis method is used to analyze the power spectrum of the profile by Fourier transformation. The resolution is (a) 11 nm for the 800 kfci track, (b) 12 nm for 900 kfci, and (c) 11 nm for 1000 kfci.

(4) Batch Fabricated High-resolution MFM Tips for Both Soft and Hard Magnetic Materials

Magnetic force microscopy (MFM) is one of the most often used techniques in the investigation of surface magnetic structures. The MFM probe consists of a very sharp magnetic tip mounted on a soft cantilever. When the probe scans over the sample, the force acting on the tip due to the stray field emanating from the sample is detected. The resolution of the MFM images is determined by the tip's size and magnetic properties. An ideal MFM tip should have a small physical size for the magnetic material at the very end of the tip, which defines the minimum detectable magnetic feature size. To obtain detectable interaction between the small size tip and the sample with an adequate signal-to-noise ratio, a sufficient magnetization of the magnetic material at the end of tip is required. Materials with higher saturation magnetization, M_s, are preferred. Another important parameter is the coercivity (H_c) of the MFM tip. If it is lower than the magnetic stray field of the sample, the magnetization direction of a MFM tip will be changed or moved during measurement. As a result, the magnetic images are difficult to explain. The MFM detects the long-range interactions that include not only the interaction between the sample and the magnetic materials near the tip, but also from the extended area of the tip. This makes the MFM images more difficult to interpret and reduces the MFM image resolution. The stray field from the extended area can be reduced by modifying the tip shape or by developing tip coating techniques. In this work, we show batch-fabricated high-resolution FePt-coated MFM probes with high H_c, high M_s and low stray field. We also present the MFM images of a garnet (soft) and a permanent magnet (hard) taken with the same tips.

The tips are made from commercial silicon micro-machined cantilevers with spring constants of 1-5 N/m, resonant frequencies of 70-89 kHz, and quality factors of about 200 in air. The thin FePt alloy films were made by dc magnetron sputtering. The film thickness was around 20 nm. These FePt MFM tips have a H_c of about 1 T and and M_s of about 900 emu/cm³.

Fig. 7 (a) and Fig. 8 are the MFM images of a garnet and a permanent FeNdB magnet using the same MFM tip. Fig. 7(b) is the MFM image that uses a commercial MFM tip.

In summary, we showed that the batch fabricated FePt coated MFM tips can be used to scan both soft and hard magnetic samples.



Fig. 7 MFM images of a garnet in the same area using (a) a new developed FePt MFM tip, (b) a commercial tip.



Fig. 8 MFM images of a permanent FeNdB magnet using a newly developed FePt MFM tip. (a) The domain pattern of the surface perpendicular to c-axis (b) The domain pattern of the surface parallel to the c-axis

(5) Microcantilever Torque Magnetometry Study of Patterned Magnetic Films

The study of small, defined magnetic structures has attracted much attention due to interest in both technological applications and fundamental research in micromagnetism. Microcantilever torque magnetometry (MTM) is a promising new experimental technique for measuring such small magnetic features. One of the challenges of using this technique is to place the sample on the cantilever. In this work, we develop a new process for preparing a patterned magnetic film on cantilever and show a primary result for magnetic interactions in a paired magnetic bar measured by MTM.

The process of patterning the magnetic film on the cantilever is the following: (a) deposit a multilayer Au (200 nm)/Cr (10 nm) on the cantilever, (b) patterning using focused ion beam (FIB) milling, (c) magnetic film deposition through a mask, and (d) a lift-off process. The magnetic hysteresis loops of the patterned dots arrays are measured by MTM at ambient conditions. Fig. 9 shows the measured hysteresis loop for a dot array with a dot diameter of 500 nm and center-to-center distance of 2 μ m. The vortex state is formed at 26 kA/m, and the single domain state happens at -49 kA/m.



Fig. 9 A hysteresis loop for a 50 nm-thick $Ni_{80}Fe_{20}$ array of 100 dots with a diameter of 500 nm and center-to-center distance of 2 μ m.

In order to understand the magnetic interaction between a pair of bars, we prepared a sample as shown in Fig. 10. A 7 μ m × 7 μ m × 30 nm Ni₈₀Fe₂₀ film was put on the top left corner of the MTM cantilever. The Ni₈₀Fe₂₀ film was then patterned by a FIB workstation into two single 7 μ m × 3.5 μ m × 30 nm bars by cutting a 50 nm gap in the center of the film [Fig. 10(a)]. After the measurement, the top bar was removed with FIB milling and just left a single 7 μ m × 3.5 μ m × 30 nm bar on the cantilever [Fig. 10(b)].

Magnetic properties of the patterned $Ni_{80}Fe_{20}$ films were characterized using a MTM at room temperature and in air. All measurements were done with a magnetic field applied in plane and perpendicular to the axis of the cantilever, as marked in the top of Fig. 10(a). Fig. 11 shows the magnetic hysteresis loops for the single and paired 7 μ m × 3.5 μ m × 30 nm Ni₈₀Fe₂₀ bars. For the single bar, the magnetization reversal occurs around the coercivity field of -1.23 kA/m. It is correlated to the domain wall propagating quickly through the bar. The smaller jump in magnetization at - 4 kA/m may be caused by the annihilation of the small domain structure at the edge of the film. For paired bars, the magnetization reversal occurs at -1.3 kA/m and -1.7 kA/m. The first jump at a field of -1.3 kA/m corresponds to the reversal of one of the paired bars. The second jump at a field of -1.7 kA/m corresponds to the reversal of one of the paired bars and less than that of both paired bars indicates magnetostatic interaction exists between the closely paired bars and that is consistent with our micromagnetic simulations.

In summary, we develop a process to pattern a magnetic film on the MTM cantilever and by using MTM, we studied the magnetic interaction between the close small size and shaped-defined magnetic elements with high resolution.



Fig. 10 (a) Double 7 μ m × 3.5 μ m × 30 nm bars patterned with focused ion beam (FIB) on the 7 μ m × 7 μ m × 30 nm Ni₈₀Fe₂₀ film with a gap of 50 nm between adjacent bars. (b) Single 7 μ m × 3.5 μ m × 30 nm bar after removing the top bar with FIB.

Fig. 11 Magnetic hysteresis loops obtained with a microcantilever torque magnetometer of the single 7 μ m × 3.5 μ m × 30 nm Ni₈₀Fe₂₀ bar and same size paired bars with a gap of 50 nm.

(6) Torsion Oscillator Magnetic Field Sensors

Microcantilever torque magnetometry (MTM) is a promising new experimental technique for measuring such small magnetic features due to its high sensitivity. In this work, we develop a new sensitive magnetic sensor based on MTM that can detect nT range field change under ambient conditions and we expect pT sensitivity with further improvement. The principle of the MTM sensor , $\tau = \vec{m} \times \vec{H}_t$, is shown in Fig. 12(a). τ is the torque that applied to the cantilever. The magnetic sample is magnetized (\vec{m}) by an applied magnetic field along the sample plane. A small ac torque magnetic field is applied perpendicular to the sample plane (\vec{H}_t) by a solenoid coil that excites the cantilever at the resonance frequency. Our current MTM can detect a magnetic moment change of 10⁻¹² Am² with a $H_t = 10^{-4}$ T, that is, a change in $\tau = 10^{-16}$ N·m can be measured. As shown in Fig. 12(b), the lock-in signal is plotted as a function of time as the magnetic flux is stepped in 16 sec intervals. The mark shows that a 55 nT change in magnetic field has about 15 mV output from the lock-in amplifier.



Fig. 12 (a) A schematic diagram of the set-up of the torsion oscillator magnetic field sensor, and (b) the lock-in signal is plotted as a function of time as the magnetic flux is stepped in 16 sec intervals. The mark is a 55 nT change in magnetic field.

(7) Imaging Magnetic Noise Sources in Magnetic Recording Heads

A detailed understanding of noise characteristics is essential for the design of a high signal-to-noise ratio (SNR) reader sensor. We intend to correlate the microstructure to the source of magnetic noises for improving the magnetic stability of the recording heads. A dynamic magnetic sensitivity mapping (MSM) system was designed to image the magnetic noise sources in sub-micrometer sized recording heads. A nanometer sized magnetic force microscopy (MFM) tip was used to apply a well-defined, localized magnetic field on the air bearing surface (ABS) of the head. For a certain area position of the free layer with incoherent rotation of the magnetic moment, this localized magnetic field will cause magnetic instability in the head, and this instability will show up as electrical noise in the output signal. Because most of the noise related to magnetic domain fluctuation is dominant in the low frequency region, our study concentrates on the spatial characterization of the noise source in a frequency range of 20 kHz to 60 kHz. Recording the average amplitude of the noise spectrum due to the excitation in the measured frequency range as a function of the tip's position, the location of the magnetic noise source can be identified. Magnetic noise images have been obtained by our system for some giant magnetoresistance (GMR) and tunneling magnetoresistance (TMR) recording heads. Noise MSM images of some unstable recording heads clearly show the spatially uneven noise

The noise mapping images of stable and unstable TMR heads with positive and negative tip magnetization in the frequency range of 20 kHz-60 kHz are shown in Fig. 13. For head I, the noise maps [Fig. 13(a) and (b)] show that there is no additional noise due to magnetic tip excitation whether under a positive or a negative tip magnetization state. However, the noise map for head 2 [Fig. 13(c)] shows that there is considerable noise having spatial variation. A magnetic noise increase is detected only during tip scanning over the left side of the sensor and a small amount of noise is observed in the right part of the sensor. Upon reversing the tip field direction, this localized magnetic noise disappears [Fig. 13(d)].



Fig. 13 MSM images for the TMR head I (stable) (a) under a positive magnetic field and (b) under a negative field; and for magnetic TMR head 2 (unstable) (c) under a positive magnetic field and (d) under a negative field.

(8)Dependence of Noise in Magnetic Tunnel Jjunction Sensors on Annealing Field and Temperature

Sensor noise is a crucial parameter in low-field applications and the minimum detectable field of the sensor is limited by their intrinsic noise. In order to resolve this issue, we investigated the low frequency noise in magnetic tunnel junctions (MTJs) which were configured into 64 element symmetric bridges as shown in Fig. 14. They were annealed in the temperature range from 265°C to 305°C and magnetic fields up to 7 T, either in helium or hydrogen environments. It can be seen from Fig. 15 that the noise level of the MTJ annealed in hydrogen gas at 0.5 T external magnetic field is reduced by two times at low frequency (at 1 Hz) compared with the original sample (which was annealed at 285°C and in 0.5 T field) and the noise level of the MTJ annealed under hydrogen gas at 7 T is reduced by three times at low frequency (at 1 Hz) compared with the original sample. The source of noise in MTJs can be magnetic or non-magnetic in origin, which can be distinguished by measuring in different magnetic fields. There is about an order of magnitude reduction of the low frequency noise spectrum when the MTJ is measured with a 10 mT bias magnetic field. (In this magnetic field the magnetization of the free layer is parallel to that of the pinned layer.) The reduction of the noise at low frequency might due to the removal of some of defects and the pinning sites change in the free layer as well as the pinning layer during the annealing in high magnetic field. By optimizing the annealing temperature in hydrogen, we may further improve the noise floor at low frequency in the future, in other words, improve the minimum detectable field of future sensors at low frequency.



Fig. 14 The picture of the MTJ bridge with a junction size of 5 μ m x 7.5 μ m. Inset is an enlarged picture of those MTJs



Fig.15 Noise spectrum of MTJ bridge with junction size $10 \ \mu m$ x 15 μm annealed at 265°C in fields of 0.5 T and 7 T in a hydrogen environment.