VI.3 Metrology for Fuel Cell Manufacturing

Eric Stanfield
National Institute of Standards and Technology (NIST)
100 Bureau Drive, MS 8211
Gaithersburg, MD 20899-8211
Phone: (301) 975-4882; Fax: (301) 975-8291
E-mail: eric.stanfield@nist.gov

DOE Technology Development Manager:
Pete Devlin
Phone: (202) 586-4905; Fax: (202) 586-9811
E-mail: Peter.Devlin@ee.doe.gov

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Project End Date: October 1, 2009

Objectives

Metrology for Fuel Cell Manufacturing Subprojects

- P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance (Fiscal Year 2008 DOE Funding, Completion FY 2008)
  - Objective: Develop a pre-competitive knowledge base of engineering data relating fuel cell performance variation to manufacturing process parameters and variability.
- P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control (FY 2008-FY 2009 DOE Funding)
  - Objective: Identify and evaluate the capability and uncertainty of commercially available non-contact, high-speed scanning technologies for applicability to bipolar plate manufacturing process control.
- P3 Optical Scatterfield Metrology for Online Catalyst Coating Inspection of Proton Exchange Membrane Fuel Cell (PEMFC) Soft Goods (FY 2008 NIST Funded, FY 2009 DOE Funded)
  - Objective: Evaluate the suitability of Optical Scatterfield Metrology (OSM) as a viable measurement tool for in situ process control of catalyst coatings.

Technical Barriers

These projects address the following technical barriers from the Manufacturing R&D and Fuel Cells sections of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

(F) Low Levels of Quality Control and Inflexible Processes

(B) Lack of High-Speed Bipolar Plate Manufacturing Processes

Contribution to Achievement of DOE Manufacturing Milestones

This project will contribute to achievement of the following DOE milestones from the Manufacturing R&D section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- **Milestone 1:** Develop prototype sensors for quality control of MEA manufacturing. (4Q, 2011)
- **Milestone 2:** Develop continuous in-line measurement for MEA fabrication. (4Q, 2012)
- **Milestone 7:** Develop manufacturing processes for graphite resin, natural flake graphite, and metal plates. (4Q, 2010)

Accomplishments

- P1 Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance.
  - Fabricated and verified (in-house) through dimensional inspection ten experimental cathode fuel cell flow field plates with intended dimensional/geometrical perturbations following our 2^{4-1} factorial design-of-experiment plan – June 2008.
  - Validation of in-house fuel cell performance testing capability – July/August 2008.
- P2 Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control.
  - Identified, procured, and have begun evaluating the capability and uncertainty of two commercially available structured light based non-contact, high-speed scanning probes for in situ manufacturing process control measurements of carbon and metallic bipolar plates.
- P3 Optical Scatterfield Microscopy for Online Catalyst Coating Inspection for PEMFC Soft Goods.
  - From consultation with membrane electrode assembly (MEA) manufacturers, national labs, and academia; we secured the technical guidance (critical parameter identification) and sample preparation support needed for our project.

– Demonstrated sensitivity of OSM to Pt catalyst density differences (different Pt/C weight percentages), thus potential sensitivity to catalyst loading (mg/cm^2) when combined with a, yet to be determined, (non-destructive, non-contact, high-speed) thickness measurement and if the Pt distribution throughout the layer can be assumed homogeneous.

NIST Project 1 - Cause-and-Effect: Flow Field Plate Manufacturing Variability and its Impact on Performance

Introduction

Based on a workshop organized by the Center of Automobile Research and NIST in December 2004, industry bipolar plate manufacturers identified a need for engineering data that relates geometric bipolar plate tolerances to fuel cell performance. This need is in response to pressure by fuel cell designers to produce lower cost plates. As such, plate manufacturers are being forced to consider potential quality related trade-offs to achieve desired cost targets. To justify these trade-offs, manufacturers are questioning the relevance of stated tolerances on dimensional features of bipolar plates; thus expressed a desire for published engineering data relating performance and dimensional quality of the plates that can be used as a reference when making these decisions. In response to the identified need, this project was conceived in 2004 and funded through the NIST Advanced Technology Program Intramural Competition for a period of three years (FY 2005-FY 2007). In 2008 funding was provided through DOE to bring this project to a successful completion by the end of FY 2008.

Approach

The main premise of this project is to fabricate cathode plates with intentional perturbations to identified dimensional features, then systematically substitute these plates with the original equipment manufacturer plates in an existing single-cell fuel cell design and through polarization curve measurements determine what, if any, impact these perturbations have on fuel cell performance. Furthermore, if a particular perturbation has an impact on performance then we aim to correlate the performance change with the magnitude of the perturbation. Finally, at the conclusion of this project, publish the results.

Results

In order to determine the effect of dimensional feature tolerances on fuel cell performance we have:

- Selected a reference fuel cell design.
- Developed a measurement inspection method to dimensionally evaluate the quality of bipolar plate features (refer to Figure 1).
- Reverse engineered the plate design used in the reference fuel cell.
- Optimized the reference cell design to maximize performance measurement repeatability.
- Dimensionally evaluated carbon and metallic sample plates made by commonly used manufacturing methods to quantify typical quality levels of various geometric parameters to establish a baseline and range.
- With industry guidance, chose four parameters to intentionally perturbate (side-wall taper, side-wall channel direction straightness, bottom channel direction straightness, and variation in channel width).
- Developed a statistically-based experimental design.
- Developed and demonstrated the capability to competently perform single-cell testing (refer to Figure 2).
- Became proficient at machining high-quality carbon-based plates.

Conclusions and Future Directions

In 2008, we have completed fabrication and inspection of all experimental plates and are awaiting the completion of a fuel cell test stand repair and upgrade prior to conducting the performance measurements with the experimental plates. We are optimistic that our performance testing and related publication will be completed by October 1, 2008.

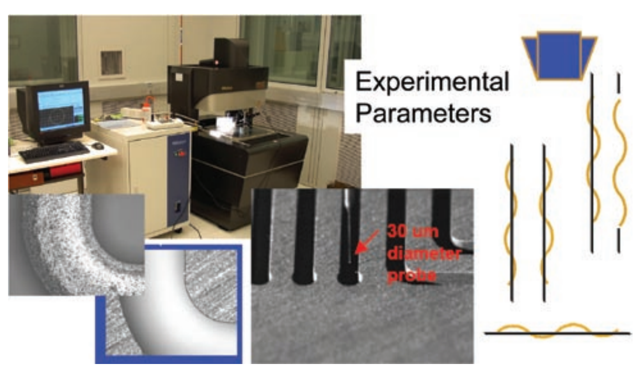

FIGURE 1. Dimensional Inspection Capability and Graphical Representation of Experimental Parameters

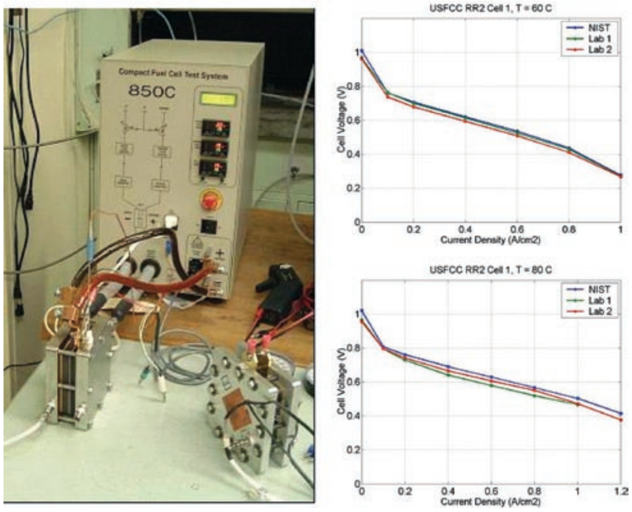

FIGURE 2. PEMFC Single Cell Fuel Cell Test Stand and Preliminary Test Results from the U.S. Fuel Cell Council (USFCC) Single-Cell Task Force Round Robin #2

NIST Project 2 - Non-Contact Sensor Evaluation for Bipolar Plate Manufacturing Process Control

Introduction

Based on input from numerous bipolar plate manufacturers, we initiated a project in the summer of 2007 to identify and/or develop non-contact, high-speed measurement techniques for dimensional measurement of general bipolar plate dimensions and their flow field, alignment, and manifold features. Additionally, measurement technology identified and/or developed must be capable of measuring metallic and carbon-based materials, must be suitable for in situ integration in a manufacturing line, and must demonstrate accuracy of five micrometers or better. To get this project moving, we invested our own discretionary money by way of procurement of two of the most promising non-contact structured light sensors identified through our market research. Since this time and DOE's 2008 funding of the project, we have developed two different phases of testing and begun our extensive investigation into the performance of these two sensors.

Approach

From our research experience in the mid- to late-1990s testing non-contact structured-light sensors for the measurement of threaded gages, we immediately knew what the limitations might be for this particular application. With the knowledge that these types of sensors can have difficulty with highly-reflective surfaces and large incident angles we developed an initial phase of testing that focused on these parameters. If the sensors passed this initial evaluation then they would be mounted on a test bed which consisted of a three-axis machine tool that would enable precise movements. The second phase of testing would enable us to evaluate feature amplitude and location measurement accuracy. Finally, the third phase (conducted on the same test bed with a few modifications) will enable us to evaluate the accuracy as a function of scan speed. The second and third phases will provide the performance data necessary to optimize the evaluation time as a function of desired dimensional accuracy, which is absolutely critical with regards to minimizing product cost. Development of these three phases and their associated test beds enables us to evaluate any non-contact sensor that we wish, with the hopes of identifying the quickest method of evaluation while ensuring accuracy. Along with this effort we will be developing a check standard(s) and a calibration protocol(s) that will establish traceability (a common contractual requirement).

Results

From the inception of this project to date, we have accomplished the following:

- Procured two non-contact, structured light-based laser spot sensors both working on somewhat of a different application of triangulation. Probe 1 is a simple 30 um diameter spot laser triangulation probe and Probe 2 is a 30 um diameter spot holographic based laser triangulation probe.
- Interfaced both the sensors to a personal computer to allow for automatic operation and data collection.
- Tested both sensors using our Phase 1 protocol that focuses on reflectivity of material and incident angle.
  - Probe 1 (Figure 3) showed excellent sensitivity and repeatability on very shiny and diffuse surfaces. It also demonstrated excellent repeatability and linearity measuring very shiny surfaces up to an incident angle of about 60º.
  - Probe 2 showed similar results as Probe 1; however had difficulty with the very shiny surface.
- Setup and characterized the three-axis machine tool and engineered a versatile mount for attaching the probes to the machine Z-axis; all necessary for Phase 2 testing (Figure 4).
- Obtained very promising Phase 1 and initial Phase 2 results for Probe 1 with regards to amplitude accuracy (probe sensing direction) and feature width or length (scan direction) when measuring flow field features of a carbon-based fuel cell plate (Figures 5 and 6).

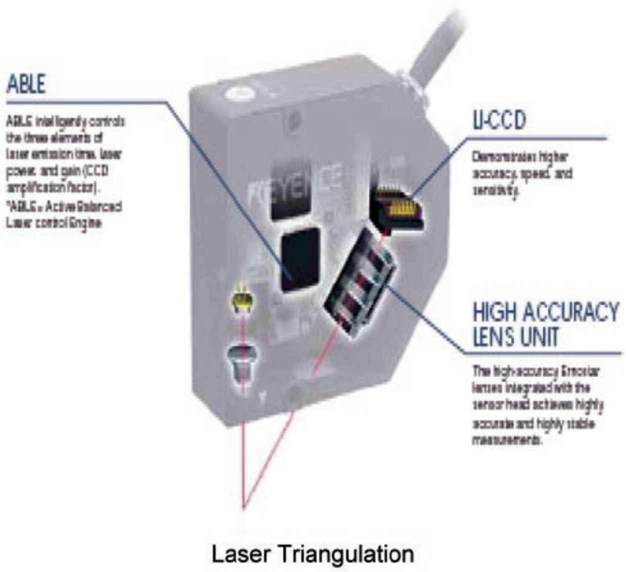

FIGURE 3. Principle of Operation for a Simple Laser Spot Triangulation Probe

FIGURE 4. Phase 2 Testing: 3-Axis Machine Tool with Probe 1 Mounted to Machine's Z-Axis

Conclusions and Future Directions

To date, we have made significant progress developing the means by which to appropriately evaluate any non-contact sensor and in the actual evaluation of the two procured probes. Testing for both probes will continue even though Probe 2 had difficulties with very reflective surfaces during Phase 1. It is our belief that the shiny artifact (steel gage block) we chose for Phase 1 testing sets the extreme limit for reflectivity and based on the matte finish of several sample metallic plates we believe further testing may be of benefit. Specifically, the manufacturer of Probe 2 claims a sampling rate almost seven times faster than that claimed by the manufacturer of Probe 1 (0.3 ms/point versus 2 ms/point). These claims in conjunction with repeatability will be evaluated in Phase 3.

For the next year, to complete all testing of the two probes, we intend to procure a precision variable speed precision linear stage that can be mounted to the stage of the machine tool providing us the capability to test accuracy as a function of scan speed (the stage

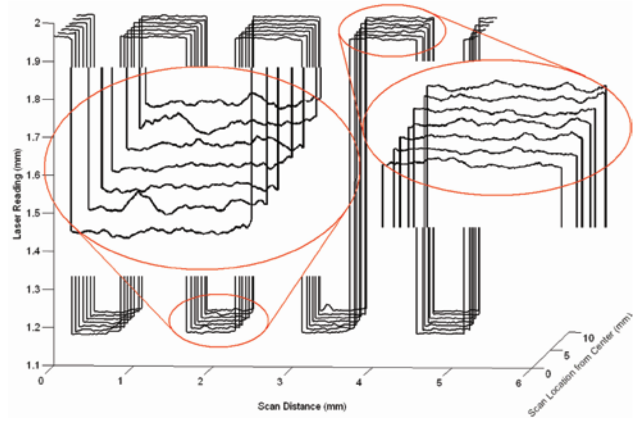

FIGURE 5. Phase 2 Quasi 3-Dimensional Plot of Multi-Line Scan Data using Probe 1 (Note: Scan data in direction orthogonal to the channel width not yet acquired and integrated)

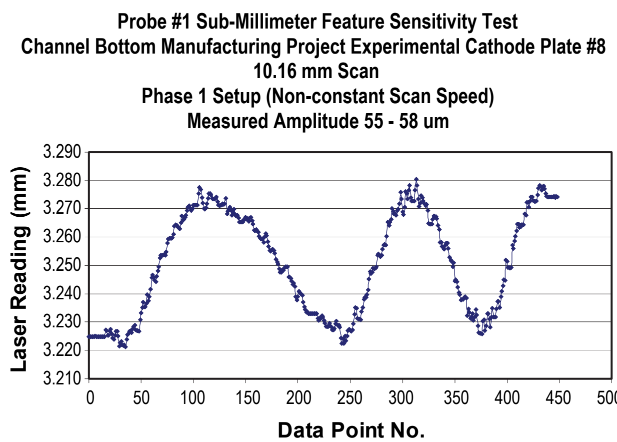

FIGURE 6. Phase 1 Linear Plot Showing Sensitivity to In-Channel 50 um Amplitude Features (Note 1: Characterized test plate provided from Manufacturing Cause-and-Effect Project) (Note 2: X-Axis data is not correlated to a particular dimensional scale)

of the machine tool is limited to 1 to 1.5 mm/s). Also during the next year, we would like to procure a laser triangulation-based line probe (25 mm x 30 um) and, due to the ever increasing resolution of digital cameras, we would like to investigate the potential of these to make photogrammetry measurements. For clarification, photogrammetry measurements are dimensional measurements derived from the camera resolution using images from three cameras each with a known and different orientation with respect to the part. This can and is done currently but the question is whether or not the resolution of these top end cameras has reached a point that can support the desired accuracy for this application.

NIST Project 3 - Optical Scatterfield Metrology for Online Catalyst Coating Inspection of PEMFC Soft Goods

Introduction

The need for high-speed, in situ process control measurement techniques for controlling the quality of the platinum-based catalyst layer has been identified during numerous DOE sponsored and non-DOE sponsored workshops. Current techniques for the various parameters of interest are extremely expensive and not easily amendable to in situ application in a manufacturing production line. The Precision Engineering Division within the Manufacturing Engineering Laboratory has years of expertise with a technology identified as OSM [1], specifically its development as a process control tool for the semiconductor industry. This technique is a combination of the best attributes of traditional optical metrology and scatterometry. More precisely, this technique takes traditional optical metrology beyond the Rayleigh criteria (resolvability of features) and focuses on the complex optical signatures of subwavelength size features, where the response can be sensitized by varying the illumination angle, varying the illumination source frequency, and application of various image analysis algorithms. This technique is a comparative tool, thus far as applied in this application and must be correlated to measurements of the parameter of interest using reference metrology such as scanning electron microscopy (SEM). As such, the development for new applications requires research being done to optimize the OSM tool and establishing the appropriate reference metrology tool, along with the development of the appropriate analysis algorithms for both. Please refer to the stated reference for more technical details on the technique.

Prior to this project this technique has only been applied to very discrete uniform structures, such as, those typically encountered in the semiconductor industry. Because of this, the application of the OSM technique to random subwavelength structures has long been a goal but it does present significant challenges. The underlying principle is that even random subwavelength structures exhibit some type of repeatable optical signature and that changes in these signatures can be correlated with the various manufacturing parameters of interest to control the quality of their product.

This project was initiated in FY 2008 and funded solely through an internal exploratory solicitation. At the end of the FY we hope to evaluate the progress of this effort to determine if further research can provide significant benefits. If it can, it is the hope that further research can be conducted and funded under our interagency agreement with DOE.

Approach

Due to our lack of experience and knowledge with regards to the technical details related to the fabrication and performance of catalyst layers, this project depends heavily on support from MEA manufacturers, as well as, other experts in the field. From the beginning, we actively engaged MEA manufacturers and industry experts in an effort to identify the critical parameters of the catalyst layer and to provide sample packages that vary these parameters to enable us to conduct a sensitivity study of proposed technique. Our hope is that we can obtain sample packages from various sources in an effort to investigate numerous critical parameters. With this, it is our assumption that if, for example, we are able to test seven critical parameters and our technique is sensitive to five of these, then application of the technique will still be useful.

Results

From the inception of this project to date, we have accomplished the following:

- Developed a working relationship with the Hawaii National Energy Institute (HNEI), where they have been providing technical assistance and prepared several sample packages.
  - These sample packages include preparation of the catalyst layer on a Teflon®-coated fiberglass reinforced Kapton transfer decal. Samples include: carbon black, various Pt loadings using a 20% Pt/C mixture, and various Pt loadings using a 50% Pt/C mixture.
  - Based on recent results, HNEI is in the process of preparing numerous samples covering the spectrum from 15% to 60% Pt/C to enable us to

precisely calibrate the sensitivity of our tool to the Pt/C weight percentage mixture.

- Signed non-disclosure agreements with two MEA manufacturers (sample package details have yet to be determined), while actively pursuing other agreements.
- Utilized the expertise of our relationship with Los Alamos National Laboratory researchers.
- Initiated potential cooperative effort discussions with the National Renewable Energy Laboratory who is doing similar but different MEA manufacturing and performance research. Their research regarding thickness measurement of MEAs and their defect investigation may provide extremely useful to the demonstration of our technique.
- Performed initial SEM investigations of catalyst layers applied to a carbon cloth-based gas diffusion layer (GDL) and applied directly to the membrane to study the effects of underlying substrate (Figure 7).
- Through the use of our focused ion beam milling capability on our SEM, we began to study the cross-sectional platinum particle distribution homogeneity within the catalyst layer.
- Using ion beam milling, we created fiducial marks on the HNEI samples to identify specific locations that can be located using both SEM and OSM tools.
- Performed extensive measurements on the carbon black, 20%, and 50% HNEI samples using the OSM tool and developed numerous analysis algorithms. One of the algorithms demonstrated promising sensitivity to the amount of Pt present in each of the three types of samples; blank carbon, 20% Pt/C, and 50% Pt/C (Figure 8).

Conclusions and Future Directions

Through our initial interactions with manufactures, fuel cell designers, and industry experts we learned in general what critical parameters of the catalyst layer are believed to affect performance. The level of technical detail needed to perform a measurement evaluation reinforces the need for cooperative agreements with manufacturers since the specific information is not in the public domain. Furthermore, our technique could prove quite useful with regards to identifying defects; however this can not be tested without industry knowing at what size or density the defects affect performance.

We have demonstrated that the OSM technique is sensitive to the Pt/C weight percentage of catalyst layer; however this is not a measure that alone can be a measure of platinum loading, the critical parameter of interest with the HNEI sample package. It is our hope that a surface measurement with the OSM technique along with a catalyst layer thickness measurement and the assumption of a homogeneous platinum distribution throughout the cross-section can result in a meaningful measure of platinum loading. Before researching a means of making a non-destructive, in situ coating thickness measurement we are going to attempt to demonstrate the efficacy of aforementioned idea by way of a detailed SEM study. If this proves to be a legitimate possibility then we will examine the thickness measurement issue. In the meantime, we will work with the manufacturers for which we have an agreement to identify other critical parameters, acquire related sample packages, and investigate the sensitivity of the OSM tool to these parameters.

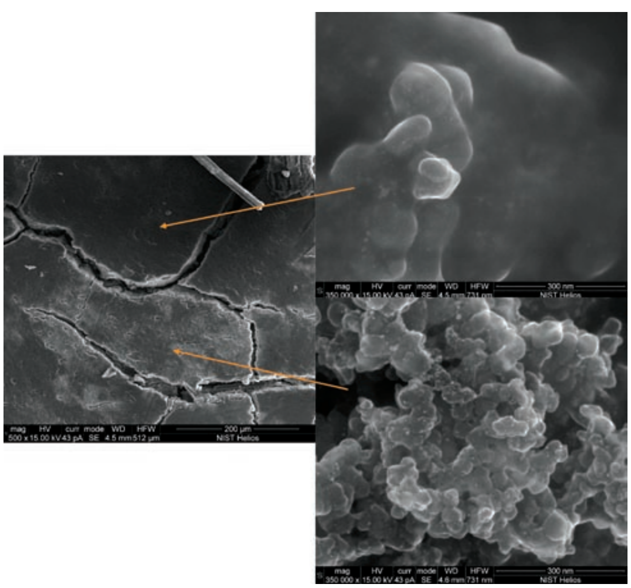

FIGURE 7. SEM Secondary Electron Image of Cloth GDL

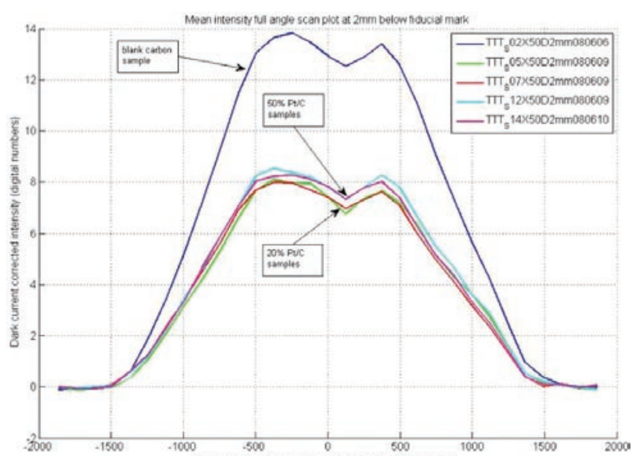

FIGURE 8. OSM Image Analysis using Mean Intensity Algorithm Showing Repeatable Signature Differences. Top Profile – Carbon Black (1 Sample – 4 Locations), Middle Profiles - 50% Pt/C (2 Samples – 4 Locations Each), Bottom Profiles – 20% Pt/C (2 Samples – 4 Locations Each)

Complete technical details of our studies to date are available upon request in the form of an internal exploratory report submitted to our laboratory management. Prior to dissemination of this report will need to be modified to protect proprietary information.

FY 2008 Publications/Presentations

1. DOE AMR June 13, 2008.

References

1. R. M. Silver, B.M. Barnes, R. Attota, J. Jun, M. Stocker, E. Marx, and H. Patrick, "Scatterfield Microscopy for Extended Limits of Image-Based Optical Metrology," Applied Optics, Vol. 46, No. 20 (2004).

Disclaimer

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