Hydrogen Compatible Materials Workshop

November 3rd, 2010
Research, Engineering, and Applications Center for Hydrogen
Sandia National Laboratory, Livermore, CA

Introduction:

On November 3rd, 2010, Sandia National Labs hosted a workshop focused on hydrogen compatible materials and components. The goals of the workshop were two-fold, 1) to identify gaps in hydrogen compatible materials R&D, and 2) to develop international R&D pathways that address the identified R&D gaps.

This workshop document contains the following sections:

- **Workshop Agenda** - The topics and schedule for the workshop.
- **Morning Presentation Overview** - A brief summary of the content from each presentation in the morning workshop session.
- **Process for Gap Identification and Prioritization** - A description of the mechanics of the afternoon working meeting on gap identification.
- **Gaps Identified** - This section summarizes the primary output from the afternoon working meeting, i.e., enumerating and assigning priority to gaps. The gaps are organized into three categories: Data and Phenomenology, Technology Development, and Codes and Standards.
- **R&D Pathways** - Proposed R&D frameworks for addressing the highest-priority gaps.
- **Appendix** - A list of workshop participants with affiliations and contact information.

Workshop Agenda:

The morning agenda of the workshop was structured to provide contemporary perspectives from the U.S. Department of Energy (DOE), Original Equipment Manufacturers (OEMs), Standards Development Organizations, industrial gas companies, research labs, and universities. The afternoon agenda was an interactive working meeting focused on gap identification and research road-mapping. The final agenda is shown below:

7:45 – 8:15 AM	Coffee and Continental breakfast
8:15 – 8:30 AM	Welcome, Introductions and Charter (Brian Somerday)
8:30 – 8:45 AM	Scott Weil/**DOE** - FCT program activities
8:45 – 9:15 AM	Katsuhiko Hirose/**Toyota** - automotive industry, automotive codes & standards
9:15 – 9:45 AM	Glenn Scheffler/**SAE** - automotive codes & standards
9:45 – 10:00 AM	Coffee Break
10:00 – 10:30 AM	Steve Hoffman/**Air Products** - H2 production, distribution, dispensing industry
10:30 – 11:00 AM	Jader Furtado/**Air Liquide** - H2 production, distribution, dispensing industry

11:00 – 11:30 AM	Chris San Marchi/**Sandia** - H2 Compatible Materials R&D
11:30 – 11:45 AM	Petros Sofronis/**Univ. Illinois** - International Institute for Carbon-Neutral Energy Research
11:45 AM – 12:45 PM	Lunch
12:45 – 3:00 PM	Gap identification
3:00 – 3:15PM	Coffee Break
3:15 – 4:15PM	Input consolidation, clarification, prioritization
4:15 – 5:30PM	International R&D and Code Development Road-mapping

Morning Presentation Overview:

Scott Weil from the DOE Fuel Cell Technologies Program (FCT) provided an overview of the Program activities in Fuel Cells, Production, Delivery, Storage, and Codes and Standards. Dr. Weil focused on the challenges facing delivery in 3 Technology Areas 1) Pipelines, 2) Tube trailers/storage vessels, and 3) Compressors/Dispensers. Pipelines (both existing and new) must mitigate the impacts of hydrogen embrittlement while reducing capital costs with the use of new alloy compositions and methods of joining. In the area of tube trailers/storage vessels, cost must be reduced via use of lower cost/high performing fiber overwrap. The hydrogen capacity must be increased through enabling higher pressure. Safety must be enhanced to promote greater/more rapid commercial acceptance. Finally, in the area of compressors and dispensers, component durability must be enhanced. Capital cost must be reduced though use of new materials. Dr. Weil described the cross-cutting nature of hydrogen effects in materials research as similar needs are present for H2 production, H2 storage, and fuel cell technology. All of these areas also require uniform methods of materials/component testing and codification.

Katsuhiko Hirose from Toyota provided the automotive industry perspective for hydrogen technologies. Dr. Hirose described the FCV commercialization plans in Japan. Japan has very aggressive targets for 2015 and 2020 for the deployment of fuel cell vehicles. Toyota sees FCV as the only viable technology for mid to long-range applications that meet performance and cost requirements. New materials are critical for FCVs. Dr. Hirose stated that the “end of stone age was not due to the lack of stone”. New materials lead the technological innovation and enable changes in the energy society. Environmental pressures are driving the industry to find new materials and enable a more sustainable energy society.

Glenn Scheffler (a contractor for DOE FCT) described the Society of Automotive Engineers (SAE) perspective on automotive codes & standards. Dr. Scheffler described how systems-level performance-based requirements provide “final verification” of system safety prior to deployment, although these can be challenging when performed in hydrogen gas. Appropriate hydrogen compatibility methodologies can aid designers in the selection of pressure-bearing materials and greatly simplify the verification protocols and thereby reduce time/cost. In addition to providing “pedigree” for component selection and type-test approval, component specifications can provide a basis for making component substitutions without having to repeat all systems-level, performance-based tests. Dr. Scheffler described the specific challenges faced in defining requirements for hydrogen compatibility of pressure-bearing materials. First, test protocols for identifying materials for hydrogen applications need to be defined. Second, the design limits need to be specified for materials already demonstrated to be acceptable of hydrogen service. Third, a performance-based test method must be defined to demonstrate the acceptability of materials in specific designs.

Steve Hoffman of Air Products provided an industrial gas company perspective. Hydrogen materials challenges he highlighted include A) hydrogen embrittlement resulting in reduction of strength, ductility, or toughness, B) the need for high strength materials despite the tendency for increased susceptibility to hydrogen embrittlement, C) the need to understand materials behavior in hydrogen and CNG blends. In summary, Air Products seeks better materials to address short-term and long-term solutions that further advance product safety, reliability, performance, and lower long term costs. Additionally, more hydrogen embrittlement data for high-pressure hydrogen is needed, including data for accelerated fatigue in cyclic service. Air Products also sees need for continued Codes & Standards development, and education is essential to ensure the safe use of hydrogen as a fuel is adhered to across all suppliers and users.

Jader Furtado provided the Air Liquide perspective on materials needs for H2 production and distribution. Dr. Furtado described the market demand for new technologies to increase the efficiency and reduce the cost of deploying hydrogen to consumer applications. Innovation in materials is driven by, the demand for new packaging technologies (i.e., higher storage capacity), the need for new delivery systems (including fueling stations, cylinder connectors, etc.), and new operating conditions (for example, increased pressure, load cycles, etc). Dr. Furtado outlined the challenges faced when qualifying the performance of materials in a hydrogen environment. Specific challenges include, A) materials must be tested at the same conditions (pressure and temperature) as expected in the usage environment, e.g., it is important to ensure that metallic materials are tested in contact with hydrogen gas, B) the testing methods developed (and resulting standards and codes) must be harmonized. Dr. Furtado suggested that a round-robin testing program among international laboratories could be the basis for such harmonization.

Chris San Marchi provided the Sandia perspective on gaps in the area of Hydrogen Compatible Materials. Dr. San Marchi divided the gaps into three categories as follows: 1) R&D gaps - lack of fundamental understanding of mechanisms and behavior, 2) Code gaps – test method development and harmonization, and 3) Technology Development gaps. Specific to R&D gaps, Dr. San Marchi described the need to understand the sensitivity of hydrogen embrittlement to material composition, environmental variables, and gas purity. In addition, the influence of internal vs. external hydrogen is poorly understood. Also, acceleration of test methods must be based on real measured behavior and/or mechanism understanding. In the area of code gaps, clarity is needed to establish effective hydrogen embrittlement parity between similar materials with slightly differing compositions. Additionally, appropriate fracture mechanics and fatigue life test methods must be developed and harmonized. Finally, Dr. San Marchi highlighted two general technology development gaps. First, low cost and/or high-strength materials are needed. Second, simplified testing methods for materials and components enable the deployment of new technologies.

Petros Sofronis from the University of Illinois, Urbana-Champagne ended the morning session with a presentation describing the new International Institute for Carbon-Neutral Energy Research (I^2CNER), a program funded by MEXT in Japan and led by Dr. Sofronis. The Institute includes a research program in the area of Hydrogen Structural Materials led by Dr. Brian Somerday. The goals of the Hydrogen Structural Materials division are to, A) understand fundamental interactions of hydrogen with metals and alloys, and B) design materials with known resistance to hydrogen embrittlement. Programs such as I^2CNER will be leveraged to coordinate international research in hydrogen effects in materials.

Process for Gap Identification and Prioritization:

Effective gap identification requires gathering input from all stakeholders followed by prioritization by the community. The result of this process is a relevant list of high-priority activities that can be organized into a coherent R&D pathway. Gaps were solicited for three categories: 1) Data and Phenomenology, 2) Technology, and 3) Codes and Standards. Topic leaders were identified for each category to describe and organize the content as it was generated from the attendees. Upon completion of the gap generation from the attendees, the category leaders reviewed each gap and grouped like items. Each attendee was then provided with 5 sticker dots to help prioritize the gaps identified. Following prioritization, the Category leaders reviewed the results with the community and discussed the implications. Although originally planned, the developing and coordinating future research plans based on the prioritized gaps was deferred.

Gaps Identified:

Gaps were solicited for three categories: 1) Data and Phenomenology, 2) Technology Development, and 3) Codes and Standards. Gaps are listed in order of priority as indicated by attendees.

Data and Phenomenology

This category addresses fundamental material behavior and associated mechanisms as well as data needs. The gaps identified are as follows in order of priority as determined by the workshop participants:

DP-1	Measurements of mechanical properties of structural metals in high-pressure hydrogen gas, in particular fatigue properties (both crack initiation and crack propagation). The dependence of fatigue properties on the following variables must be established: load-cycle frequency, R ratio (i.e., mean stress), cyclic loading waveform, gas purity, hydrogen pressure, and temperature. Materials test methods must be optimized (e.g., reduce cost) and results must be interpreted reliably. A notably high proportion of the workshop participants recognized this gap as a priority. (15 votes)	✪✪✪✪ ✪✪✪✪ ✪✪✪✪ ✪✪✪

DP-2	Database for properties of structural materials in hydrogen gas. This gap was identified based on the need for readily accessible data for designers. This database would be an open source, and results included in the database would originate from reliable or accredited institutions. The database must include not only material property values but also details on test procedures and material characteristics related to the properties. It was specifically noted that mechanical property data are needed for a variety of steels and aluminum alloys. A notably high proportion of the workshop participants recognized this gap as a priority. (12 votes)	✪✪✪✪ ✪✪✪✪ ✪✪✪✪
DP-3	Influence of welds on hydrogen compatibility of structures. A notably high proportion of the workshop participants recognized this gap as a priority. (11 votes)	✪✪✪✪ ✪✪✪✪ ✪✪✪
DP-4	Fundamental research studies are needed to address fatigue crack initiation in hydrogen gas. One unresolved issue is whether structural metals exhibit an endurance limit (i.e., stress threshold for unlimited life) in hydrogen gas. Basic mechanisms for fatigue crack initiation in hydrogen gas must be established for structural metals such as steels and aluminum alloys. (4 votes)	✪✪✪✪
DP-5	Structural material qualification for pressures and temperatures relevant to portable power applications. (4 votes)	✪✪✪✪
DP-6	Understanding the combined effects of temperature variations (particularly during fast filling) and hydrogen gas on structural materials in fuel cell vehicle tanks. (3 votes)	✪✪✪
DP-7	Understanding the combined effects of temperature variations (in particular, low temperatures near -60 ^{o}C) and hydrogen gas on structural materials in dispensing components. (3 votes)	✪✪✪
DP-8	Hydrogen effects in non-metals (e.g., polymers), including aging, wear, and structural changes. (3 votes)	✪✪
DP-9	Defining appropriate methods for measuring material properties in particular components. For example, the relevant fatigue properties (i.e., crack initiation or crack propagation) must be identified for specific components. (2 votes)	✪✪
DP-10	Compatibility of structural materials with gas blends (e.g., hydrogen/natural gas). (2 votes)	✪✪

DP-11	Effects of residual stress on fracture (e.g., fatigue cracking) of structural metals in hydrogen gas. (1 vote)	✪
DP-12	Understand the effects of collision-related damage on the design life of fuel cell vehicle components.	

Technology Development:

Technology development gaps are those missing portions of the technology space that prevent greater penetration of hydrogen systems. The gaps identified are as follows in order of priority as determined by the workshop participants:

TD-1	High-strength, low-cost materials for long-life hydrogen service. This is a universal need for gaseous hydrogen fuel components. For the most part, service experience is limited to low-strength materials or limited-life components. Relevant service experience with a wider range of materials is needed, especially higher strength metals. New materials may need to be developed specifically for hydrogen service. Comprehensive testing of higher strength materials is needed to provide engineers with a broader selection of materials for hydrogen service. (8 votes)	✪✪✪✪ ✪✪✪✪
TD-2	Reliable hydrogen compressors, primarily associated with production/distribution/dispensing but impacts all technology areas that store hydrogen fuel as high-pressure gas. The current practice for many hydrogen fueling applications relies on storage of high-pressure (≥35 MPa) gaseous hydrogen. Hydrogen compressors are commonly identified as the "weak link" in dispensing high-pressure gaseous hydrogen on a high cycle basis. Several participants identified the need to enable materials selection for hydrogen compressors as well as to invest in the design and development of compressing technology for hydrogen. (5 votes)	✪✪✪✪ ✪
TD-3	Non-destructive evaluation or NDE. Better NDE tools to monitor the health of hydrogen systems are needed. This includes new sensing technologies, improved diagnostics, real-time monitoring, etc. (5 votes)	✪✪✪✪ ✪

TD-4	Life assessment and leak-before-break criteria. Criteria for assessing and validating leak-before-break need to be better defined. Establishment of leak-before-break is a critical safety requirement for hydrogen systems, particularly systems in the hands of consumers. The criteria may be different for different applications (stationary, automotive). In addition, methods and criteria for life assessment need improvement, which likely depends on improved NDE methods (see gap above). (4 votes)	✪✪✪✪
TD-5	Material specifications for hydrogen service. Existing materials specifications can be very broad and may lead to a wide range in hydrogen compatibility behavior for the materials that meet the same specification. The classic example is AISI 316, which displays a range of hydrogen embrittlement susceptibility due to alloy content, but which also has many global variations. New materials specifications are needed to better control materials for hydrogen service and harmonize the materials that are used in the global marketplace. This is also a Data and Phenomenology gap since the materials variables that affect hydrogen compatibility are not known for all classes of materials. (2 votes)	✪✪
TD-6	New material design methodology. This gap is related to a Data and Phenomenology gap for developing methods of design that quantitatively incorporate elements of both crack initiation and crack propagation individually. Such methodologies should improve designs and clarify design margins. (1 vote)	✪
TD-7	Definition of operating conditions for portable power. This gap was identified in discussion but did not receive any votes in the technology development context. However, it received several votes in the code requirement context as “design requirements”. Arguably, design requirements make the gap a technology gap, from which spring the code requirements. See code gap below on “design requirements for portable power”.	
TD-8	Use of passivating additions to gaseous hydrogen that would mitigate the deleterious effects of hydrogen on structural metals	
TD-9	Quantification of shock and vibration in hydrogen systems	
TD-10	Selection criteria for various alloys	

Codes and Standards:

Code requirement gaps are generally associated with protocols, definitions and acceptance criteria. The gaps identified are as follows in order of priority as determined by the workshop participants:

CS-1	Testing protocols for materials evaluation. This gap includes three slight variations on the same concept: develop and optimize universal testing protocols for characterizing the mechanical properties of materials in hydrogen environments, in particular properties that may be used in design. This includes accelerating tests (implicitly testing rates and fatigue frequency) as well as testing at very high pressure. These ideas represent some of the motivations for developing the CSA CHMC1 document (Compressed Hydrogen Materials Compatibility). (10 votes)	✪✪✪✪✪ ✪✪✪✪✪
CS-2	Engineering acceptance criteria. There is significant interest in establishing clear definition(s) of reliability for hydrogen service components, such that a coherent and harmonized set of engineering criteria can be applied to components in hydrogen service. (9 votes)	✪✪✪✪✪ ✪✪✪✪
CS-3	List of acceptable materials. This gap was identified in the context of motive fuel cell systems and perhaps stimulated by the example established in the SAE J2579 standard of accepting certain materials. A major challenge here is that most materials exhibit some degree of degradation in hydrogen gas, and the "compatibility" of a material of construction can be dictated by the design of the component (stress level, safety factors, etc) and the environment in which it is used. Solely composing a list of materials may not be an adequate materials qualification strategy. (5 votes)	✪✪✪✪✪
CS-4	Design requirements for portable power. The lack of design requirements for portable power (non-motive fuel cell systems) applications was noted by a number of participants. This was identified as a gap in code activities, but is also coupled to the technology. Definition of conditions for portable power may facilitate development in this area. Could this mean identification of a targeted application to aid development of products? And thus illuminating the code gap? (4 votes)	✪✪✪✪

CS-5	International high-pressure standards. This code gap was motivated in the framework of production/distribution/dispensing. A number of stakeholders noted the lack of international codes for handling of high-pressure gases (>35 MPa). In order for hydrogen to become widely accepted as a fuel, specific code language pertaining to the appropriate delivery of high-pressure gaseous hydrogen is needed, particularly in locations that are “closer to the public” and where high-pressure flammable gases have not been extensively used, such as warehouses (lift trucks), office spaces (off-grid fuel cell power), airports (tugs), construction sites (portable lighting), etc. (4 votes)	✪✪✪✪
CS-6	Codes and standards variations in the context of production/distribution/dispensing. This gap was not clearly articulated, but appears to be related to inspection, perhaps the authority for inspecting hydrogen dispensing systems. There was also discussion of costs, however, by definition cost should not be a primary consideration for safety codes and standards. (4 votes)	✪✪✪✪
CS-7	Performance-based testing protocols. There is interest, in the context of motive fuel cell systems, to develop and validate appropriate performance-based system testing protocols. There is much debate among the stakeholders in the automotive sector about the appropriate testing requirements for full-scale systems (e.g., pneumatic versus hydraulic testing, rates of testing, etc.) (2 votes)	✪✪
CS-8	Testing methodology for hydrogen storage cylinders. There is interest in re-evaluating the existing code requirements for testing materials for hydrogen storage cylinders, such as ISO 11114-4. There is concern that the test methods in the ISO document are not sufficient for hydrogen fueling applications, because the test methods may not be robust and the test methods do not consider fatigue failure. (1 vote)	✪
CS-9	Classification of risk and probability of failure (e.g., fire and tanks for ground storage)	
CS-10	Fire resistance of composite vessels	
CS-11	Definition of acceptable design life for dispensing systems	

R&D Pathways

Research, development, and harmonization activities will be coordinated through the Research, Engineering, and Applications Center for Hydrogen (REACH) at Sandia National Labs. REACH is a consortium-based approach to complex hydrogen materials challenges that leverages unique facilities, programs, and personnel with international partners. The following R&D frameworks are proposed to address the four highest-priority gaps identified from the workshop:

- DP-1 and CS-1. Measurements of mechanical properties of structural metals in high-pressure hydrogen gas, in particular fatigue properties (both crack initiation and crack propagation) and testing protocols for materials evaluation. This research activity focuses on establishing the detailed data trends for hydrogen-assisted fatigue crack initiation and propagation of structural metals in hydrogen gas. The foundation of this experimental effort is conducting complex material-property measurements in high-pressure hydrogen gas. Such measurements require specialized laboratory testing capabilities, which exist at a limited number of institutions worldwide. This reality coupled with the anticipated size of the test matrix (e.g., numerous mechanical and environmental variables must be explored for each material) requires participation from multiple laboratories around the world. The need to involve multiple research institutions thus requires ***formulating international partnerships***. Additional ***partnerships must be developed with industry stakeholders*** to ensure technologically relevant materials and testing conditions are included in the research effort. Mechanical-property measurements from this activity can be archived in a proposed database (i.e., gap item DP-2). Another output from this activity is optimized fatigue test methods that balance data reliability and test efficiency (i.e., test rate).

- DP-2. Database for properties of structural materials in hydrogen gas. The objective of this activity is to create an open-source database that is structured for particular consumers, e.g., hydrogen containment component designers. The nucleus of the database is mechanical property data for a wide range of structural materials in hydrogen gas. In addition to mechanical property values, the database must include associated details on test procedures and material characteristics. Participants in this activity must include specialists in database software tool design, one or more institutions to host the database, and recognized technical experts to screen and validate candidate data. The ideal sources for populating the database are reliable and accredited materials testing institutions that can commit to contributing results. Since existing mechanical property data for structural materials in hydrogen gas are incomplete (for example, see the Technical Reference for Hydrogen Compatibility of Materials, www.sandia.gov/matlsTechRef), a ***significant effort involving multiple institutions worldwide is required to populate the database*** (e.g., gap item DP-1).

- DP-3. Influence of welds on hydrogen compatibility of structures. The objective of this activity is to experimentally characterize the properties of welds and evaluate their effect on hydrogen containment structures. Welds are a concern since their mechanical properties can vary from the base metal and fabrication processes for critical hydrogen infrastructure components are expected to involve welding, e.g., long-distance transport pipelines and local distribution piping. One of the challenges in characterizing properties of welds is that the material microstructure is inhomogeneous, e.g., regions of the weld (fusion zone, heat-affected zone) have distinct microstructures and each region can exhibit microstructure gradients. Consequently, material test specimens must sample multiple regions in the weld. Despite efforts to sample multiple regions in the weld, data may exhibit scatter and establishing relationships between microstructure and mechanical properties can be difficult. Because of the inherent variability in weld microstructures, it is important to validate analytical structural life prediction methods that employ measured mechanical properties of welds by conducting tests on full-scale or model welded structures. For example, analytical fatigue life predictions of welded stainless steel hydrogen distribution piping must be validated by quantifying the design life of full-scale or model welded piping structures that are pressure cycled in hydrogen gas. The need for validation is motivated not only by variability in weld microstructures but also the presence of ill-defined residual stresses in welded structures. ***This research activity requires partnerships between institutions having specialized high-pressure hydrogen testing capabilities*** (i.e., both mechanical property measurements in high-pressure hydrogen gas as well as pressure cycling of structures using hydrogen gas) ***and industry stakeholders*** that can supply technologically relevant weld samples and welded structures.

- TD-1. High-strength, low-cost materials for long-life hydrogen service. Structural materials for hydrogen containment components have multiple attributes that must be considered in design, e.g., cost and performance. The objective of this activity is to optimize materials for specific hydrogen containment applications. For example, hydrogen containment components that demand superior hydrogen compatibility are often fabricated from austenitic stainless steels, however these materials can be cost prohibitive since certain alloying elements (e.g., nickel) are expensive. In this case, research activities for stainless steels may focus on eliminating expensive elements such as nickel but without significantly compromising mechanical properties in hydrogen gas. As another example, high-performance centrifugal hydrogen compressors require structural materials having high strength. However, structural metals generally become less compatible with hydrogen as the material strength increases. The research challenge here is to enhance material strength while maintaining acceptable hydrogen compatibility. These efforts to optimize materials require detailed understanding of the fundamental mechanisms of hydrogen-assisted fracture and the associated material characteristics governing the mechanisms. This research activity requires participation from materials scientists with expertise in establishing relationships between material microstructure and mechanical properties. An additional dimension in determining these structure-property relationships is incorporating the interaction of hydrogen with the material microstructure. Institutions involved in this activity must have modern, high-resolution instruments for characterizing material microstructures and hydrogen-material interactions. As a result, ***partnerships with world-class research universities are essential***. Other necessary partnerships include ***material manufacturers*** that have research laboratories, so that tailored materials can be created and supplied. The involvement of material manufacturers also enhances the prospect for scaling laboratory products to commercial production.

Appendix - Workshop Participants

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