# OPERATIONAL EXPERIENCE OF CERAMIC HONEYCOMB PASSIVE AUTOCATALYTIC RECOMBINER AS A HYDROGEN MITIGATION SYSTEM

#### Chang Hyun Kim, Je Joong Sung, Sang Jun Ha

Central Research Institute, Korea Hydro and Nuclear Power Co., Ltd. 25-1 Jang-dong, Yuseong-gu, Deajeon, 305-343, Rep. of KOREA changhyun.kim@khnp.co.kr; jejoong.sung@khnp.co.kr; sangj.ha@khnp.co.kr

## **Phil Won Seo**

Department of Research & Development, Ceracomb Co., Ltd. 312-25 Deuksan-dong, Asan-si, Chungcheongnam-do, 336-120, Rep. of KOREA seopw@ceracomb.co.kr

## ABSTRACT

Ensuring the containment integrity during a severe accident in nuclear power reactor by maintaining the hydrogen concentration below an acceptable level has been recognized to be of critical importance after Fukushima Daiichi accidents. A passive autocatalytic recombiner (PAR) has been considered as a viable option for the mitigation of hydrogen risk because of its passive operation for the hydrogen removal. All Korean nuclear power plants were equipped with various types of PARs as a safety enhancement measure after Fukushima accident. Under the long-term exposed condition by airborne substances known as volatile organic compounds (VOC), it has been reported that the catalyst elements show a degraded performance for hydrogen removal. To identify the effect of VOCs on the performance of catalysts, a series of tests have been performed into three groups; (a) the measurement of start-up delay time for hydrogen removal, (b) the measurement of hydrogen removal rate and (c) VOC component analysis to identify airborne substances adsorbed on the surface of catalysts. This paper describes the detailed test results of Pt/TiO<sub>2</sub> coated ceramic honeycomb PARs, which were installed in 18 operating units in Korea. The hydrogen depletion rates and their start-up delay time have been measured using a total of 376 catalyst samples from eight (8) nuclear power plants after one overhaul period of exposure to containment air. The hydrogen depletion rate and start-up delay time are key important parameters of PAR performance model in the hydrogen control analysis for the determination of the capacity and the location of PARs. The type of airborne substances has been identified through a qualitative GC/MS (gas chromatograph/mass spectrometer) method from selected samples from five (5) plants. Through a series of tests on the functionality of PAR under the normal operation of an overhaul period, the volatile organic compounds (VOCs) delay the start-up for hydrogen removal by poisoning or blocking of the catalytic surface. Although the measured delay times were well within 30 minutes in the condition of 3 vol.% of hydrogen, 60 °C of temperature and 1.5 bar of pressure, the delay time would increase in proportion to the exposure time to containment air. The tests also showed that the hydrogen depletion rates are not affected by VOC accumulation on the catalyst surface due to its volatile nature at high temperature by exothermic catalytic reaction. The volatile organic substances adsorbed on the catalyst surface are estimated mainly from paints, lubricants, glues, insulations and oils etc.

#### **KEYWORDS**

Passive Autocatalytic Recombiner (PAR), Volatile Organic Compounds (VOC), Hydrogen Mitigation System, Start-up Delay Time

#### 1. INTRODUCTION

Ensuring the containment integrity during a severe accident in nuclear power reactor by maintaining the hydrogen concentration below an acceptable level has been recognized to be of critical importance after Fukushima Daiichi accidents. Although there exist various hydrogen mitigation measures, a passive autocatalytic recombine (PAR) has been considered as a viable option for the mitigation of hydrogen risk under the extended station blackout conditions because of its passive operation characteristics for the hydrogen removal [1]. As a post-Fukushima action item, all Korean nuclear power plants were equipped with PARs of various suppliers. The capacity and locations of PAR as a hydrogen mitigation system were determined through an extensive analysis for various severe accident scenarios [2]. For some plants, dual hydrogen mitigation systems were equipped with a combination of newly installed PARs and the existing igniters that each system has 100% of full capacity for hydrogen control for postulated severe accident conditions. Among a total of 28 units Korean nuclear power plants (including 4 units under construction), a Pt/TiO<sub>2</sub> coated ceramic honeycomb PAR supplied by Ceracomb Co. Ltd. has been installed in 18 operating plants and several units has reached a first overall period since its first installation in 2013.

The PAR makes use of a catalyst to convert hydrogen ( $H_2$ ) and oxygen ( $O_2$ ) into water vapor and heat. The heat of reaction creates a natural convective flow through the recombiner, eliminating the need of pumps or fans to transport new hydrogen to the surface of the catalyst. In spite of an advantage of its passive operation, there have been concerns about adverse effects on the performance of PARs by potential deactivators (chemical poisons and physical inhibitors) [3,4,5]. PARs are required to perform their safety function not only after exposure to potential contaminants during operation, but also in an accident environment that may contain various gases or aerosols that are potentially poisonous to the PAR catalyst elements. The Ceracomb also has performed various tests and demonstrated that its performance degradation of hydrogen removal capacity is within 25% in severe accident conditions such as fission product poisons, aerosols, cable burns, carbon monoxide, etc. However, its performance under the long-term exposed condition to containment air has not been fully investigated because Ceracomb PARs have no operation experience in normal operation conditions.

Under the long-term exposed condition by airborne substances, it has been known that the catalyst shows a delayed response for hydrogen removal [6]. These airborne substances are known as volatile organic compounds (VOC) that adsorb on active sites of the catalyst surface thus making them unavailable for catalytic reaction to proceed. As a result, the recombiner would require either a higher hydrogen concentration, or a higher temperature, or both, to start the hydrogen recombination reaction, compared with the catalyst in as-new condition. VOCs could be originated from solvents, lubricants and paints, etc. depending on the maintenance history in the containment during overhaul periods and the release of fumes from such products when they are heated upon plant restart. The catalyst functionality under the long-term exposed condition of VOCs could be the start-up delay time for catalyst reaction and its hydrogen depletion (removal) rate because these parameters directly affect the results of hydrogen control analyses in design basis and severe accident conditions. The catalyst functionality should be verified up to sufficient periods of plant operation and be compared with the parameters on the PAR functionality used in the hydrogen control analysis. Therefore, with increasing time after installation of PARs (exposure time to containment air), the VOC effects during normal nuclear power plant operation will play a more important role in PAR maintenance [7].

This paper describes operational experiences of  $Pt/TiO_2$  coated ceramic honeycomb PARs after an overhaul period of normal operation. To investigate the effect of airborne substances in containment air, a series of tests focused on the start-up delay time and the hydrogen removal rate has been performed with catalyst samples taken from eight (8) operating power plants. The analysis for VOC components has been performed to identify airborne substances adsorbed on the surface of catalysts.

## 2. TEST METHOD

#### 2.1. Pt/TiO<sub>2</sub> Ceramic Honeycomb PAR

Figure 1 shows an illustrated view of  $Pt/TiO_2$  coated ceramic honeycomb PAR that has been installed in eight (8) operating units. This type of PAR has been developed and supplied by Ceracomb Co. Ltd. [8]. The Ceracomb PAR consists of a stainless steel housing equipped with catalysts inside the lower part of the housing. The PARs are installed with floor mount type or wall mount type in the containment and its structures are designed to meet the seismic requirements of each plant. Air and hydrogen mixture flows from bottom of the PAR to the exit openings at the upper part of PAR. The housing is designed to have chimney effects so that the heat generated in the catalytic reaction in lower part of the housing can promote a strong driving force for natural convective flow and to protect the catalyst from the direct impinge of containment spray. There are three different sizes of PAR according to the number of the catalyst. The specifications of the Ceracomb PAR are summarized in Table 1.



Figure 1. Pt/TiO<sub>2</sub> Coated Ceramic Honeycomb Passive Autocatalytic Recombiner

	Small-Size	Medium-Size	Large-Size	
Weight (kg)	42.1	75.8	144.3	
Width (cm)	37.8	72.5	142.8	
Depth (cm)	34.3	36.5	36.5	
Height (cm)	100	100	100	
No. of Catalysts	4	8	16	
H <sub>2</sub> Depletion Rate $(g/sec)^{a}$ (4 %-H <sub>2</sub> , 60 °C, 1.5 bar)	> 0.2 g/sec	> 0.4 g/sec	> 0.9 g/sec	

**Table 1. Specifications of Ceracomb PAR** 

a) Required hydrogen depletion rate in the technical specification for PAR purchase of Korean NPPs.

Different types of catalytic recombiners have been supplied by various PAR suppliers such as AREVA, CANDU Energy, and NUKEM (formerly NIS). AREVA and CANDU Energy utilized plate type catalysts while NUKEM invented a specialized cartridge containing pellet type catalysts. In the present Pt/TiO<sub>2</sub> coated ceramic honeycomb PAR, a cubical catalyst with a honeycomb microstructure has been used to

increase the surface area for the reaction. The catalyst is manufactured by coating a mixture of  $TiO_2$  and Pt on the supporting structure of the ceramic honeycomb of 35 CPSI (cell per square inch). Figure 2 shows an illustrated view of ceramic honeycomb catalyst. The dimensions of the standard honeycomb catalyst are 15 cm by 15 cm with the height of 5 cm. A protected metal frame is used to protect the catalyst because the ceramic catalyst is fragile and vulnerable to impact.



Figure 2. Ceramic Honeycomb Catalyst

## 2.2 Test Facility

The VOC effect tests have been performed using the PAR performance test facility (PPTF). The PPTF comprises a carbon steel pressure vessel with the internal volume of 12.5 m<sup>3</sup> (a cylindrical shape with 3.3 m in height and 2.2 m in diameter). It was constructed to perform performance tests in various conditions of pressure, temperature, humidity, hydrogen concentration and chemical water spray. Figure 3 shows PPTF with a measurement type and locations in the pressure vessel. Inside of the vessel, mixing fans, spray nozzles and electrical heaters are installed. At the center of the vessel a test PAR is located. A small-sized PAR with four (4) catalysts is used as a test PAR. Gates are equipped at the PAR entrance and exit to prevent air and hydrogen from being in contact with the catalyst surface before the test starts.



Figure 3. PAR Performance Test Facility (PPTF) with Measurement Types and Locations

### 2.3 Test Methods

The function of the catalyst should be inspected periodically using a specially designed device during every plant outage period. In case of the present ceramic honeycomb PAR, at least a quarter of the entire catalysts are tested in every outage period since their installation in the containment. The catalysts are tested in single arrangement under the pre-determined flow and temperature of air and hydrogen mixture by measuring the temperature rise of air-hydrogen mixture between inlet and outlet of test device. Key parameters of catalyst functionality are considered as the start-up delay time and hydrogen removal rate which are directly related to PAR modeling in the hydrogen control analysis to determine the capacity and locations of PAR system [2]. Under the VOC-affected conditions, its functionality is hard to identify through the in-service testing method because the start-up delayed time and the hydrogen removal rate are defined under the natural convection conditions.

Therefore, a number of catalysts are withdrawn out of containment after the exposure to containment air during an overhaul period of normal operation and their functionality is tested in the PAR performance test facility (PPTF) under the natural convection conditions. Table 2 shows the number of catalysts taken from various plants for VOC effect tests performed in 2014. Further tests for other plants are scheduled according to their outage schedules. The VOC effect tests are performed into three groups; (a) the measurement of start-up delay time for hydrogen removal, (b) the measurement of hydrogen removal rate and (c) VOC component analysis to identify airborne substances adsorbed on the surface of catalysts. Four (4) catalysts are withdrawn from one PAR considering the installed location in the containment so that the tested catalysts be distributed uniformly throughout containment area in order to avoid local effects of the test results. The exposure time of catalysts to the containment air includes the normal operation time of ~ 18 months and the plant outage time that depends on the outage schedule of each plant.

Plant ID	Plant Type	Test Date - (yy/mm)	No. of catalysts (No. of Tests)			
			Delay Time	Depletion Rate	VOC Component	
А	PWR <sup>a</sup>	2014.04	8 (2)	8 (2)	1	
В	PWR <sup>a</sup>	2014.09	20 (5)	12 (3)	-	
С	PWR <sup>a</sup>	2014.11	28 (7)	20 (5)	-	
D	PHWR <sup>b</sup>	2014.07	20 (5)	12 (3)	1	
Е	PWR <sup>c</sup>	2014.11	84 (21)	20 (5)	1	
F	$PWR^d$	2014.12	40 (10)	12 (3)	1	
G	PWR <sup>d</sup>	2014.06	40 (10)	20 (5)	1	
Н	$PWR^d$	2014.07	20 (5)	12 (3)	-	
	Total		260 (65)	116 (29)	5	
Notes : a) 950 MWe Westinghouse 3-loop PWR c) 950 MWe Framatome 3-loop PWR		b) 700 MWe PHWR (CANDU6) d) 1,000 MWe 2-loop PWR (OPR1000)				

Table 2. Number of Catalysts for VOC Effect Tests

The start-up delay times for hydrogen removal are measured in the PPTF facility. Four (4) catalysts are mounted in a small sized test PAR housing that is the same model of the commercial PAR so that four (4)

catalyst samples are used for a test. The test PAR is installed at the center in the test vessel of the PPTF. After the test vessel is closed, mixing fans are turned on and the hydrogen is injected to a desired hydrogen concentration. Until desired conditions are achieved, gates at the PAR entrance and exit are closed in order to prevent air and hydrogen from being in contact with the catalyst surface. The start-up delay tests are performed at the initial conditions of the hydrogen concentration of 3 vol.% and temperature of 60 °C under the pressure of 1.5 bar (abs). The start-up delay time is defined as the required time for the hydrogen concentration in the test vessel to start to decrease by one percent (relative) of the initial hydrogen concentration after the hydrogen in the test vessel starts to contact the catalysts in the PAR (i.e., the gates at the PAR entrance and outlet are opened).

The hydrogen depletion rates with degraded catalysts under the normal operation environments for an overhaul period are measured using the PPTF facility. The tests are performed with the same procedure of the start-up delay time tests but with different initial conditions. The hydrogen depletion tests are performed with the initial conditions with a hydrogen concentration of 6.9 vol.% and temperature of 60  $^{\circ}$ C under the pressure of 1.5 bar (abs). The hydrogen depletion rate is calculated from the variation of the hydrogen concentration at the PAR entrance. The hydrogen depletion rate from the present tests are compared with the hydrogen depletion rate required in the technical specification of PAR purchase, which is defined as above 0.2 g/s for the small sized PAR at the conditions of 4 vol.% of hydrogen, temperature of 60  $^{\circ}$ C and pressure of 1.5 bar.

The composition adsorbed airborne substances on the catalyst surfaces is analyzed with GC/MS (gas chromatograph/mass spectrometer) method. Tests are performed by Lab Frontier Co. Ltd. using Agilent 6890 GC/5973N MSD and PT-2020D Pyrolyzer. Each catalyst is heated up in an oven and the temperature is raised up to 300 °C and 600 °C successively with a rate of 20 °C/min. The VOCs desorbed from the catalyst surface were separated continuously and their components are analyzed qualitatively with GC/MS method.

#### 3. RESULTS AND DISCUSSION

A total of 65 tests are performed with 260 sample catalysts to investigate the effect of volatile organic compounds (VOCs) on the start-up performance on the hydrogen removal. The catalyst samples are taken from eight (8) plants with four (4) different reactor types. Figure 4 shows the measured start-up delay times in conditions of hydrogen of 3 vol.%, temperature of 60 °C and pressure of 1.5 bar. These test conditions are selected because a start-up delay time is considered after the hydrogen concentration and the temperature reached at both 3 vol.% and 60 °C in the analysis of hydrogen control to determine the capacity and locations of PARs as a hydrogen mitigation system [2]. Fifteen (15) minutes of the start-up delay time are assumed in severe accident analyses while 12 hours of the start-up delay time is assumed in design basis accident analysis [9]. As shown in Fig. 4, the start-up delay times are well within 15 minutes except the plant E. The start-up delay time for plant E shows an average time of 893 sec with a standard deviation of 215 sec. The total averaged start-up delay times for all plants are estimated as 629.8 sec with a standard deviation of 228.5 sec.

In the design basis accident such as a loss-of-coolant-accident (LOCA), the hydrogen is generated gradually and the hydrogen concentration could be reached at 4 vol.% after several days without a hydrogen mitigation system after a LOCA takes places. In the analysis of hydrogen concentration in the LOCA, twelve (12) hours of the start-up delay time after the hydrogen concentration and the catalysts temperature reach at both 3 vol.% and 60 °C. Although the start-up delays of 12 hours are considered, there is a sufficient margin to maintain the hydrogen concentration below the regulatory limit of 4 vol.%. However, in the severe accident conditions, the hydrogen concentration in the containment abruptly

increases at the timing of the reactor vessel failure so that the margin for start-up delay for hydrogen removal may not be sufficient compared to the situation of a design basis accident. Our regulatory position is that the start-up delay times should be verified and compared to the assumptions used in the analysis of hydrogen control in DBA and severe accident conditions. In the case of plant E, the analysis of hydrogen control in severe accident conditions has been re-evaluated with a longer delay time of 30 minutes in consideration of the results of the start-up delay time measurement tests in 2014.



Figure 4. Start-up Delay Times after an Overhaul Period Exposure of VOCs



Figure 5. Hydrogen Depletion Rates after an Overhaul Period Exposure of VOCs

Figure 5 shows the hydrogen depletion rates after an overhaul period of exposure to VOCs in containment air. A total of 29 tests are performed with 116 catalyst samples from eight (8) plants as described in Table. 2. The test results show that the hydrogen depletion rates are much higher than the required depletion rate 0.2 g/sec that is specified in technical specification of PAR purchase in Koran nuclear power plants. A total averaged value is estimated as 0.2696 g/sec with a standard deviation of 0.0166. The measured hydrogen depletion rates of catalysts exposed to VOCs have no difference with those of new catalysts that is estimated as 0.2687 g/sec with a standard deviation of 0.0108. The recombination reaction takes place on some active sites on the degraded catalyst releasing the heat of reaction. This causes the catalyst surface temperature to increase creating a driving force for convective flow. Increase convective flow accelerates the reaction rate leading to further increase in the catalyst temperature until all the adsorbed VOCs desorb and all the active sites are free, i.e., the catalyst is fully regenerated. The same conclusion about the hydrogen depletion rate has been reported in the reference 6.

The adsorbed airborne substances on the catalyst surface are analyzed qualitatively using GC/MS (gas chromatograph/mass spectrometer) method for selected samples from five (5) plants. Various VOCs are detected and their main compounds are summarized in Table 3. It is estimated that these compounds are originated from paints, lubricant, insulation, bonds, etc., which is commonly used in the plant maintenance. Very similar VOCs are detected in plants F and G, which are the same plant type of OPR-1000. Although benzene, diethyl phthalate and heptane are commonly detected in plants A, D, F, and G, the detected volatile compounds differ from plant types so that further tests are scheduled for other plants to determine whether the type of VOC is dependent on plant itself or plant type. Some different compounds of methylstyrene and 1,3-pentadiene are detected in plants E, which shows a longer start-up delay time compared to other plants as shown in Fig. 4. Alkanes are also detected but exact types of compounds are not identified because there is no reference sample for alkane. These VOCs could be originated from a large-scale maintenance event during the outage. There was a steam generator replacement in plant E when the PARs were installed in 2013 but a direct relationship between different VOCs and the S/G replacement could not be identified because of insufficient data. Further study on type of VOCs is scheduled in next overhaul period of plant E to determine whether these different VOCs in plant E are temporary or not.

Plant ID Compounds	А	D	Е	F	G	Estimated Sources of VOCs
Benzene	0	0		0	0	Paint, Insulation, Glue
Dibutlyformamide		0		0	0	Paint, Lubricant
Diethyl phthalate	0	0		0	0	Oil
Docosane	0			0	0	Oil
Heptadecane	0			0	0	Oil
Heptane, 3-methylene-		0		0	0	Paint, Oil
Hexadecane	0			0	0	Oil
Octadecane	0			0	0	Oil
Methylstyrene	0	0	0			Paint, Insulation,
1,3-Pentadiene			0			Paint

Table 3. Major Compounds Adsorbed on the Sample Catalyst Surface

Through the present tests, the hydrogen depletion rate is not affected by VOC, but the start-up of the hydrogen removal could be delay due to VOCs depending on the types and exposure time to VOCs. In case of Ceracomb PAR, a quarter of catalysts installed in the containment are tested during every outage using a specially designed in-service test apparatus. Therefore, the experimental data of the VOC effects on catalysts performance up to at least four (4) overhaul periods are required to estimate the PAR performance degradation due to VOCs.

The Ceracomb PARs were first installed in Koran nuclear power plants in 2013. It has been a regulatory position on Ceracomb PAR that the start-up delay time and the hydrogen depletion rates should be verified periodically and compared to those assumed in the hydrogen control analysis for design basis and severe accidents because the Ceracomb PAR has no operational experience under the normal operating conditions. Therefore, the present tests have been mainly focused on the start-up delay time and hydrogen depletion rate in a given condition to validate the assumptions on those assumed in the hydrogen control analysis. It is considered that there is a sufficient margin to control hydrogen below the regulatory limit of 4 vol.% of hydrogen concentration in design basis accidents. However, in the severe accident conditions, the hydrogen in the containment abruptly increases at the timing of the reactor vessel failure. There may not be sufficient margin for hydrogen control in some severe accident scenarios if an additional start-up delay time more than 30 minutes is considered after the hydrogen concentration and the catalysts temperatures reached at both 3 vol.% and 60 °C in the present analysis method. The temperature of containment air in severe accident conditions is expected to be above or around 100 °C. It has been postulated that the temperature will be high enough to regenerate the PAR catalyst that had resided in the containment for a prolonged time period so that the PAR will promptly respond to hydrogen. Therefore, it is important to identify in which conditions the PAR will promptly react with hydrogen in such a long exposed condition to possible VOCs. To this end, further extensive tests on the catalyst performances in various hydrogen concentrations and temperatures will be performed with catalysts that had resided in various reactor containments and for various exposure times to containment air.

#### 4. CONCLUSIONS

The hydrogen depletion rates and the start-up delay time of a Pt/TiO<sub>2</sub> coated ceramic honeycomb PAR have been measured using a total of 376 catalyst samples from eight (8) operating nuclear power plants after one overhaul period of normal operation since its first installation in order to investigate the effect of volatile organic compounds (VOCs) on the catalyst functionality. The measured hydrogen depletion rate and start-up delay time were compared to those used in the hydrogen control analysis because these are key parameters in the determination of the capacity and location of PARs. The tests showed that the hydrogen depletion rates are not affected by VOC accumulation on the catalyst surface due to its volatile nature at high temperature by exothermic catalytic reaction. Through a series of test on the start-up delays using degraded catalysts, the VOC delays the start-up for hydrogen removal by poisoning or blocking of the catalytic surface. Although the measured delay times were well within 30 minutes in the condition of 3 vol.% of hydrogen, 60 °C of temperature and 1.5 bar of pressure, it is expected that the delay time would further increase in proportion to the exposure time to containment air. The type of airborne substances was identified through qualitative GC/MS (gas chromatograph/mass spectrometer) method from selected samples from five (5) plants. The volatile organic substances adsorbed on the catalyst surface were estimated mainly from paints, lubricants, glues, insulations and oils etc. It is expected that the reduction of VOC in the containment air may be a challenging work. Therefore, it is important to identify in which conditions the PAR will promptly react with hydrogen in such a long exposed condition of possible VOCs. To this end, further extensive tests on the catalyst performances in various hydrogen concentrations and temperatures will be performed with catalysts that had resided in various reactor containments and for various exposure times to containment air.

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