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Pt/CNTs-Nafion reinforced and self-humidifying composite membrane for PEMFC applications

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Abstract

A novel thin three-layer reinforced and self-humidifying composite membrane has been developed for PEMFCs. The membrane has two outer layers of plain Nafion and a middle layer of Pt/carbon nanotubes (Pt/CNTs) dispersed Nafion. The Pt/CNTs present in the membrane provides the sites for the catalytic recombination of H₂ and O₂ permeating through the membrane from the anode and cathode to produce water and improve the mechanical properties of the composite membrane at the same time. The water produced directly humidifies the membrane and allows the operation of PEMFCs with dry reactants. The electrochemical performance and mechanical properties of the composite membranes are compared with those of a commercial Nafion[®] membrane. The self-humidifying composite membrane could minimize membrane conductivity loss under dry conditions and improve mechanical strength due to the presence of the Pt/CNTs.

Keywords: Fuel cell; Self-humidifying membrane; Composite membrane; Pt/CNTs

1. Introduction

Polymer electrolyte membrane fuel cells (PEMFC) have long been suggested as the next generation power pack for electric vehicles due to their inherently high efficiency and low polluting gas emissions. In order to establish the future of PEMFCs as an alternative power source, they must have high power and minimal volume [1,2]. Perfluorosulfonic acid membranes, such as Nafion[®], used as the solid polymer electrolyte in these fuel cells, has to be humidified to provide a satisfactory proton conductivity because of the hydrophilic nature of the sulfonic acid groups attached to the polymer backbone and the necessity to hydrate the ionic clusters [3,4]. So far, to prevent drying out of the membrane and to keep it in its most conductive state, the reactant gases have traditionally been humidified before entering the fuel cell. However, external humidification of the reactant gases in PEMFCs is a burden for the fuel cell system and lowers the system's energy efficiency.

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Several methods have been proposed to remove the externally humidifying unit from the PEMFC system by endowing the electrolyte membrane with self-humidifying ability, such as impregnation of Nafion recast membranes with a small amount of nanosize Pt particles. Watanabe et al. [5,6] proposed selfhumidifying membranes with highly dispersed nanometer size Pt and/or metal oxides (such as SiO₂ or TiO₂). The Pt particles in the membrane were conceived to act as water generation sites by catalytic recombination of hydrogen and oxygen reactant gases permeating through the membrane from the anode and the cathode. Liu et al. [7] reported a self-humidifying composite membrane prepared by casting a mixture of Nafion solution and Pt/C catalyst onto a porous PTFE film. However, these methods had several disadvantages, including non-uniform distribution of Pt particles throughout the membrane and formation of an electron-conducting path via the network of dispersed Pt or Pt/C particles [8].

To avoid the possibility of a short circuit through the membrane, some double-layer composite membranes have been proposed. Yang et al. [9] proposed a thin double-layer composite membrane consisting of one layer of Pt/C catalyst dispersed recast Nafion and another layer of plain recast Nafion. The Pt/C

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particles dispersed Nafion layer and the plain Nafion layer were, respectively, on the anode and cathode sides of the membraneelectrode assembly (MEA). Although the double-layer composite membrane could avoid a short circuit of the membrane, the Pt/C particles in the membrane have the possibility to connect with those in the anode, so the Pt/C particles in the membrane have the possibility to be an extension of the electrode, which results in loss of the self-humidification function.

Numerical simulation of water management models indicates that water production associated with a current density of 1000 mA cm^{-2} is sufficient to completely hydrate a dry 50 μ m thick Nafion[®] 112 membrane in roughly 10 s [10]. Dhar [11] suggested the use of a very thin membrane, which allows the water molecules produced on the cathode to diffuse into the membrane, thereby humidifying the membrane. However, the durability of the cell assembled with a thin membrane is shortened by insufficient structural robustness.

In our previous work [12], we developed a CNTs/Nafion reinforced composite membrane. The composite membrane has many advantages: high mechanical strength, good dimensional stability upon hydration and dehydration, ease of handling, and good cell performance with thin membrane.

In this paper, we present a novel Pt/CNTs reinforced and self-humidifying composite membrane with a sandwich structure. The membrane has two outer layers of plain Nafion and a middle layer of Pt/CNTs dispersed Nafion. The Pt/CNTs in the membrane can act not only as water generation sites by catalytic recombination of hydrogen and oxygen reactant gases permeating through the membrane from the anode and the cathode to humidify the membrane directly but also as reinforced fibers to improve the membrane mechanical strength. The membrane prepared in this paper has many advantages: (1) the membrane is very thin $(25 \,\mu\text{m})$ which speeds up the backdiffusion of product water from the cathode to the anode; (2) the two plain Nafion layers on two sides can absolutely avoid the possibility of short circuit due to the electronically conducting Pt/CNTs particles; (3) the two Nafion layers separate the Pt/CNTs in the self-humidifying layer with Pt in the electrode, so the Pt/CNTs in the membrane only have self-humidification function without the possibility to become the extension of the electrode.

The novel self-humidifying and reinforced composite membrane solves the problems of the CNTs/Nafion membrane, and converts its disadvantages to advantages by using a selfhumidifying layer. The structure of the film was investigated with scanning electron microscopy (SEM) and infrared spectroscopy (FT-IR). The mechanical strength, membrane hydration and the PEMFC performance of the composite membrane were tested and compared with those of commercial Nafion[®] membrane.

2. Experimental

2.1. Pt/CNTs and membrane preparation

The purified multi-walled nanotubes used in this study were purchased from Shenzhen Nanotech Port Co, Ltd. The average diameter of the CNTs was about 40 nm, the length was around several micrometers and the purity was above 95 wt.%. For the better anchoring of the metal nanoparticles, CNTs were oxidized by refluxing in concentrated HNO₃ at 413 K for 4 h [13,14]. Pt/CNTs nanocomposites were prepared by microwave heating of ethylene glycol (EG) solutions of Pt precursor salts. As a typical process for the synthesis of Pt/CNTs, a proper volume of an aqueous solution of 0.05 M H₂PtCl₆·6H₂O was mixed with 25 ml of EG, and 0.4 ml of 0.4 M NaOH in a beaker. CNTs (0.200 g) were uniformly dispersed in the mixed solution by ultrasonic bath. The beaker was placed in the center of a microwave oven (LG WD700, 2450 MHz, 700 W) and heated for 60 s. The resulting suspension was filtered, and the residue was washed with deionized water. The solid product was dried at 393 K overnight in a vacuum oven. The loading of Pt on CNTs was about 1 wt.%.

A Nafion[®] commercial dispersion in low aliphatic alcohols was used for recasting the composite membrane. First the aliphatic alcohols in Nafion[®] dispersion were replaced by N, N'dimethylformamide (DMF). Then a proper weight of Pt/CNTs was put into the Nafion-DMF dispersion. The suspension was ultra-sonicated for 30 min to form a homogeneous dispersion, then was poured into a flat-bottom glass dish and dried at 50–60 °C for about 10 h to form a film with a thickness of 20 μ m. The thickness of the membrane can be easily handled by controlling the amount of Nafion solution. The loading of CNTs in Nafion resin was about 3 wt.%. Then the dried Pt/CNTs-Nafion membrane was peeled, intact, from the glass dish. The 5 wt.% Nafion and isopropanol (IPA) solution was sprayed onto the two sides of Pt/CNTs-Nafion membrane, resulting in a layer of 2.5 µm thick recast Nafion layer at each side. The thickness of the sprayed Nafion layer was also handled by controlling the amount of Nafion solution. Then the composite membrane was dried in a vacuum oven at 165 °C for 90 min to dry the solvent completely. The film was light black. Re-acidification of the cast film was carried out using 0.5 M H₂SO₄ solution for 60 min at 80 °C followed by two boiling water treatments (60 min each). For a comparison, a control Nafion membrane with three-layers of plain recast Nafion in the both sides and a CNTs/Nafion layer without Pt in the middle was also prepared in a similar way with the same thickness. The three-layer composites with and without Pt thus prepared are denoted hereafter, respectively, as N-Pt/CNTs-N and N-CNTs-N. The membranes produced here have a thickness of 25 µm.

2.2. Membrane characterization

2.2.1. Dimensional stability

Membrane specimens with the length of about 10 cm and the width of about 8 cm were stored at the vacuum oven of 80 °C for 24 h and the length dimension was measured before (L_1) and after (L_2) the samples were soaked in deionized water at 80 °C for 24 h. Dimensional change (ΔL) was calculated by using the following equation:

$$\Delta L(\%) = \frac{L_2 - L_1}{L_1} \times 100 \tag{1}$$

2.2.2. Water content of membranes

Samples of the membranes were weighed (W_1) after immersion in deionized water for 24 h at 80 °C. Then, samples were weighed (W_2) after drying in a vacuum oven at 80 °C for 24 h. Water content (ΔW) was calculated according to Eq. (2).

$$\Delta W(\text{wt.\%}) = \frac{W_1 - W_2}{W_2} \times 100$$
⁽²⁾

2.2.3. Mechanical strength of membranes

Samples of the membranes were dried in a vacuum oven at 80 °C for 10 h. The maximum strength was measured with a tension tester GMT 4503 (SANS, AUTO graph) at room temperature. Tensile conditions were based on Chinese Standard QB-13022-91 and samples were measured using a programmed elongation rate of 50 mm min⁻¹.

2.3. Electron microscope analysis of N-Pt/CNTs-N film and Pt/CNTs catalyst

Analysis by transmission electron microscopy (TEM) was conducted on the Pt/CNTs powders. The powders were dispersed in ethanol, using an ultrasonic bath, and then mounted on standard copper TEM grids. The cross-section surface of the composite film was observed by a scanning electron microscope (SEM). A JEM-1200EX microscope was used for TEM and SEM analysis.

2.4. Measurement of infrared spectroscopy

In order to examine whether Pt embedded in the membrane indeed recombines hydrogen and oxygen to form water inside the membrane, IR absorbance measurements have been made for the self-humidifying and as received membranes using FT-IR spectrometer. For measurements, N-Pt/CNTs-N and N-CNTs-N membranes were initially completely dried in the vacuum oven for 7 days at 95 °C. Among the dried membranes, several samples were mounted in a single cell without electrodes and operated with completely dried hydrogen and oxygen at 0.2 MPa for 2 days at 80 °C.

2.5. *Membrane electrode assembly preparation and single cell test*

The electrodes for single cell testing consisted of gasdiffusion and catalyst layers. The gas-diffusion layer was prepared by spreading an ethanol mixture of the Vulcan XC-72 carbon black and a 30 wt.% PTFE emulsion onto a Teflonized carbon paper (TGPH-060, Toray) which was then sintered at 340 °C for 40 min. The catalyst layer was prepared by applying an ethanol mixture of the Pt/C catalyst (20 wt.%, E-TEK) and 30 wt.% PTFE emulsion onto the gas-diffusion layer and then it was baked at 240 °C for 30 min and at 350 °C for 40 min. Finally, a Nafion solution (5 wt.% Du Pont) was sprayed onto the surface of catalyst layer. The PTFE content in the catalyst layer was 30 wt.%. The loadings of Pt and Nafion resin were about 0.3 and 0.4 mg cm⁻², respectively, for both the anode and cathode. Two electrodes with an active area 5 cm^2 were hot-pressed onto one piece of membrane at $140 \,^{\circ}\text{C}$ and 2.0 MPa for 1 min to form the MEA. The MEA was mounted in a single cell with stainless steel end plates and stainless steel mesh field as current collectors.

Firstly, the cells were operated with humidified reactant gases, under these conditions: humidifier temperature $T_{\text{H}_2}/T_{\text{O}_2} = 80/80 \,^{\circ}\text{C}$; cell temperature $80 \,^{\circ}\text{C}$; gas pressure $P_{\text{H}_2}/P_{\text{O}_2} = 0.20/0.20 \,\text{MPa}$. After stable performance was obtained, the cells were then operated with dry gases. Before operation with dry gases, the cells were dried overnight with dry N₂.

2.6. Gas permeability

Electrochemical method (Chrono-coulometry) was used to measure the hydrogen crossover through the electrolyte membranes [15]. The CHI 660 Electrochemical Station (CHI Corporation, USA) was used for this test. The MEA was operated with non-humidified hydrogen at the anode, and non-humidified nitrogen at the cathode. A 0.25 V voltage was applied across the cell, so that the hydrogen permeating through the membrane was oxidized electrochemically at the cathode. The flow rate of H₂ and N₂ was controlled at 10 and 30 ml min⁻¹, respectively.

3. Results and discussion

3.1. Infrared absorbance spectra of membranes

In order to examine whether water is indeed produced by the recombination of crossover hydrogen and oxygen on the Pt particles embedded in the membrane, IR absorbance measurements have been made to verify the validity of self-humidification [8,16].

Fig. 1 illustrates the infrared absorbance spectra of the membranes treated at different conditions. For measurements, all membranes were initially completely dried in the vacuum oven



Fig. 1. Infrared absorbance spectra of membranes under different conditions. All the membranes were initially dried in vacuum oven for 7 days at 95 °C. Dotted (---, N-CNTs-N) and straight (---, N-Pt/CNTs-N) lines indicate as dried membranes were mounted in a single cell without electrodes and operated with completely dried hydrogen and oxygen at 0.2 MPa for 2 days at 80 °C.



Fig. 2. TEM of the Pt/CNTs catalyst.

for 7 days at 95 °C. After drying completely, the N-Pt/CNTs-N and N-CNTs-N membranes were mounted in a single cell without electrodes and operated with dried hydrogen and oxygen at 0.2 MPa for 2 days at 80 °C. It is obviously that the absorbance peak of the O–H stretching bending near 3500 cm⁻¹ is observed only in the N-Pt/CNTs-N membrane. A typical OH stretching peak of H₂O appears at 3450 cm^{-1} . The peak at 3700 cm^{-1} is assigned to water, which is partially bonded to fluorocarbon and has one proton available for hydrogen bonding. Therefore, the IR absorbance result clearly substantiates the validity of the mechanism for water production that Pt particles embedded in the self-humidifying membrane act as a recombination center for permeated hydrogen and oxygen. At peaks below 1400 cm^{-1} , S=O and S–OH stretching peaks of the –SO₃H group and –SO₃⁻ stretching peaks are observed.

3.2. TEM photomicrograph of Pt/CNTs catalyst and SEM photomicrograph of the membrane

Fig. 2 presents TEM graph of the carbon nanotubes supported platinum catalyst. The carbon nanotubes are with a diameter of about 40 nm, many small black Pt particles are visible on the surfaces of the CNTs. The diameters of the Pt on the CNTs are uniform, ranging from 3 to 4 nm.

SEM photographs of the cryogenically fractured surface in liquid nitrogen of N-Pt/CNTs-N membrane are shown in Fig. 3. From Fig. 3a, three-layer structure of the composite membrane can be observed clearly. Layer I is neat Nafion resin layer. Layer II is Pt/CNTs dispersed Nafion layer. The white dots in layer II

represent the ends of the carbon nanotubes in the fractured surface. The distribution of CNTs in CNTs-reinforced composite is important for the mechanical properties of the composite. From Fig. 3a and b, we can conclude that the distribution of carbon nanotubes in Nafion resin is very uniform. There are no obvious agglomeration of the CNTs. Fig. 3c represents the morphology of plain Nafion resin layer. The Nafion layer is smooth and compact. From Figs. 2 and 3, we can conclude that Pt particles distribute uniformly in Pt/CNTs-Nafion layer which provide the sites as more as possible for the catalytic recombination of H₂ and O₂ permeating through the membrane from the anode and cathode, respectively.

3.3. The hydrogen permeation rate

The self-humidification occurs presumably by at least three steps. The first step is permeation of hydrogen and oxygen through the membrane, the second is internal adsorption of reactant gases on Pt particles embedded and the final step is the recombination of internally adsorbed hydrogen and oxygen on the Pt particles. Among the three successive steps, crossover by permeation of hydrogen and oxygen is in general accepted as very slow, compared to the last two steps. Therefore, high rate of crossover of hydrogen and oxygen seems to improve the water production rate in the self-humidifying membrane.

The hydrogen permeation rates of the N-Pt/CNTs-N and the Nafion[®] NRE-212 membrane (the equivalent weight and thickness of NRE-212 membrane are 1100 and 50 µm, respectively) are shown in Fig. 4. The hydrogen permeation rate of the two membranes had the same dependency on temperature: it increased as the temperature increased. This result is in accordance with the result Liu gained [17]. It can be found that the N-Pt/CNTs-N had larger hydrogen permeation rate than Nafion[®] NRE-212 membrane. This high gas permeability of the composite membrane is benefit to the self-humidification. The more gas crossover can generate more water in the composite membrane which makes the cell with the self-humidifying membrane possible to show better performance than that with the unmodified membrane. The hydrogen permeation rate of the membrane has inverse proportion to the thickness of membrane. The hydrogen permeation rates of the N-Pt/CNTs-N and commercial Nafion[®] NRE-212 are no obvious difference if we eliminate the affect of different thickness. It indicates that the composite membrane made in this paper is compact.

3.4. Dimensional stability and water content of membranes

During the actual operation of fuel cells, the dimensional stability of PEM is very important, and it is vital for keeping the stability of MEA. Table 1 shows dimensional change of N-Pt/CNTs-N composite membrane and commercial Nafion[®] NRE-212 membrane after being soaked in 80 °C water for 24 h. It is found that N-Pt/CNTs-N composite membranes could decrease dimensional change compared with the native Nafion[®] membranes. This can be attributed to the good combination between CNTs and Nafion matrix, which results in restraining the swelling of the membrane effectively.



Fig. 3. SEM of cross-sectional morphologies of the N-Pt/CNTs-N composite membrane (a and b) and the outer layer of neat Nafion (c).

3.5. Mechanical strength of membranes

Mechanical strength of membranes affects manufacturing conditions of MEA and durability of PEMFCs. Table 1 shows the maximum mechanical strength of N-Pt/CNTs-N composite membrane and Nafion[®] NRE-212 membranes. The mechanical



Fig. 4. H_2 permeation rate of the N-Pt/CNTs-N composite membrane and the Nafion[®] NRE-212 membrane with un-humidified gas.

strength of the composite membrane is better than Nafion[®] NRE-212 in spite of its thinner thickness. Good mechanical strength will make it possible for the thinner composite membrane to be used in fuel cell and improve the fuel cell performance meanwhile. Because of their one-dimensional structure with a length of several micrometers, carbon nanotubes with a good dispersion (concluded from Fig. 3) can act as bridges between the macromolecules and form weak physical cross-linking networks if there is no further treatment of orientation, thus increasing the mechanical strength of the composite membrane.

Table 1
Character of membranes

Membrane	N-Pt/CNTs-N	Nafion [®] NRE-212		
Thickness (mm)	0.025	0.050		
Water content (at 80 °C, %)	22.18	28.34		
Dimensional change (in water at 80 °C, %)	13.25	17.78		
Maximum strength (MPa)	33.42	27.20		



Fig. 5. Polarization characteristics of the MEAs fabricated with N-Pt/CNTs-N, N-CNTs-N and Nafion[®] NRE-212 membranes operated with humidified H_2 and O_2 at 0.2 MPa and 80 °C.

3.6. Performance of PEMFCs operated under fully humidified and dry conditions

The *i*-V curves for cells based on N-Pt/CNTs-N, N-CNTs-N and Nafion[®] NRE-212 operated under humidified conditions are shown in Fig. 5. Under humidified condition, the cells using the two different kinds of composite membranes show almost the same performance. The performance of the cell using N-Pt/CNTs-N is slightly better than that of the cell using N-CNTs-N, because the Pt particles in the self-humidifying composite membrane can effectively suppress the crossover of reactant gases in fuel cells, and thus lower the cathode overpotential and consequently improve the cell performance. In this regard, opencircuit voltage (OCV) values are good measurements of the fuel and oxidant crossover through the membranes. The electrodes used in both the cells were prepared identically and their activities can be assumed to be the same. Therefore, the cell with less fuel and oxidant crossover would lead to a higher OCV value. Table 2 shows the OCV of three kind of membrane under humidified and non-humidified H_2 and O_2 . It is obvious that the cell with the N-Pt/CNTs-N membrane has higher OCV values at 80 °C, indicating that the incorporation of Pt/CNTs catalyst into the Nafion membrane suppresses the crossover of H₂ and O2 gases, most probably due to the effect of the catalytic recombination of the permeating H_2 and O_2 at the Pt particles sites inside the membrane.

Fig. 6 shows the performance of the single cells using selfhumidifying and as received membranes measured under dry

Table 2

Comparison of the open-circuit voltage of cells fabricated with the N-Pt/CNTs-N, plain N-CNTs-N and Nafion[®] NRE-212 membranes at 80 °C with humidified and non-humidified H₂ and O₂

Membrane	Open-circuit voltage (V) at 80 $^\circ \text{C}$			
	Humidified gas	Non-humidified gas		
N-Pt/CNTs-N	1.013	1.010		
N-CNTs-N	0.945	0.941		
Nafion [®] NRE-212	1.006	0.992		



Fig. 6. Polarization characteristics of the MEAs fabricated with N-Pt/CNTs-N, N-CNTs-N and Nafion[®] NRE-212 membranes operated with dry H_2 and O_2 at 0.2 MPa and 80 °C.

reactant gases of hydrogen and oxygen of 0.2 MPa at 80 °C. In the case of an externally non-humidifying condition, the self-humidifying membrane cell shows considerably higher performance in comparison with that of the as received membrane cell over the entire voltage range. This result first indicates that the rate of proton transfer through the self-humidifying membrane is greater than that through the as received one. Again, considering that the rate of proton conduction is a function of water content in the membrane, the result suggests that water content in the self-humidifying membrane is higher than that of the N-CNTs-N membrane. The increased water content of the self-humidifying membrane seems to originate from the embedded Pt particles inside the membrane. The generated water can hydrate the membrane, resulting in stable operation of PEMFCs without any external humidification. But there is a decrease of fuel cell performance due to poor proton conductivity compare to external humidifying.

The relative currents generated at different cell voltages with dry H_2 and O_2 as compared to those obtained with humidified reactants are compared in Table 3 for the MEAs fabricated with N-Pt/CNTs-N, plain N-CNTs-N, and Nafion[®] NRE-212 membranes. The fraction of current density was calculated in comparison to that obtained on operating with humidified H_2 and O_2 . The fractions of current density delivered with N-Pt/CNTs-N membrane are higher than those delivered with N-CNTs-N and Nafion[®] NRE-212 membrane. These results indicate the pronounced effect of the incorporated Pt/CNTs powders in self-

Table 3

Comparison of the fraction of current density delivered by a PEMFC fabricated with the N-Pt/CNTs-N, N-CNTs-N and Nafion[®] NRE-212 membranes on operating with dry H_2 and O_2

Membrane designation	Fraction of current density at the voltage of different voltage (%)					
	0.8 V	0.7 V	0.6 V	0.5 V	0.4 V	
N-Pt/CNTs-N	91.3	92.6	92.3	88.6	85.6	
N-CNTs-N	74.8	75.2	74.1	71.8	74.5	
Nafion [®] NRE-212	71.7	72.7	73.6	71.7	74.1	

humidifying membrane and to improve the cell performance with dry reactants. The thickness of N-CNTs-N membrane is one-half of that of Nafion[®] NRE-212, so the water backdiffusion through N-CNTs-N membrane is faster than through NRE-212 membrane. This leads to that the fractions of current density delivered with N-CNTs-N membrane are higher than those with Nafion[®] NRE-212 membrane.

4. Conclusions

A novel three-layer reinforced and self-humidifying composite membrane consisting of one layer of Pt/CNTs catalyst dispersed Nafion and two other layers of recast Nafion on both sides has been made via a solution casting method. The results of SEM studies show that the Pt/CNTs particles were dispersed in the membrane homogeneously. The performance of the composite membrane have been compared with those of plain a Nafion membrane in PEMFCs using dry and humidified H₂ and O₂ reactants. The N-Pt/CNTs-N membrane shows a better performance than the N-CNTs-N membrane and Nafion® NRE-212 membrane under both dry and humidified conditions. With dry H₂ and O₂, the N-Pt/CNTs-N membrane shows about 90% of the performance obtained with the humidified reactants while the Nafion® NRE-212 membrane only shows about 72% under similar conditions. The better performance and higher OCV values of the MEAs with the N-Pt/CNTs-N membrane is attributed to the catalytic recombination of H_2 and O₂ permeating through the membrane from the anode and cathode to produce water. The Pt/CNTs could also improve the mechanical strength and dimensional stability of the composite membrane.

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