



Electrochemical sensor for determining CO and H₂O content during CO conversion for hydrogen production

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Abstract

The paper presents the results of the study on an electrochemical solid electrolyte sensor with a diffusion barrier for simultaneous measurements of carbon (II) oxide and moisture concentrations in the initial gaseous mixture for the carbon oxide conversion into hydrogen. The sensor is composed of two electrochemical cells made of commercial stabilized zirconium dioxide of the 90 mol.% ZrO₂ +10 mol.% Y₂O₃ composition. One electrochemical cell performs carbon oxide content measurements in the initial gas mixture, and another cell determines the humidity of the same gas mixture. Both electrochemical cells operate in an amperometric mode. We obtained dependences of the limiting sensor current both on the carbon oxide and on the moisture concentrations in the gas mixture, as well as the temperature dependences of the limiting currents within the range of 650–750 °C. The sensor dynamic characteristics for the measurement of carbon oxide concentration and moisture at the temperature of 700 °C were obtained. The sensor possesses a satisfactory determination rate and high reproducibility at the operating temperature of 700 °C.

Key findings

- A miniature electrochemical sensor for simultaneous determination of CO and moisture content during carbon oxide conversion for hydrogen production has been developed.
- The sensor is composed of two identical solid electrolyte electrochemical cells made of zirconium dioxide solid electrolyte with oxygen conductivity.
- The sensor allows obtaining linear dependences between the measured values of limiting currents and the concentration of the determinable component (CO and H₂O).
- The dynamic characteristics of the sensor have been obtained.
- The sensor demonstrates high reproducibility of the results and fast response to the concentration changes of the measured component.

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1. Introduction

Conversion is a process of gas transformation aimed at changing the initial gas mixture composition. Usually gaseous hydrocarbons (methane and its homologues) and carbon monoxide are converted to hydrogen and its mixtures with carbon monoxide.

The process of carbon monoxide conversion by water vapor proceeds via the reaction: $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2 + 41.19 \text{ kJ}$.

This reaction is widely used in industry and as a primary method of hydrogen production either during solid fuel gasification or as a second conversion stage of natural gas, i.e. mainly a mixture of hydrogen and carbon monoxide. The conversion reaction of carbon monoxide is reversible, exothermic, and proceeds without changes in the number of gas moles. To increase hydrogen output, the reaction should be performed at low temperatures at excess water vapor concentration.

Accompanying information

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Thermochemical, interferential and optic methods are most commonly used for carbon monoxide determination. Moreover, the thermochemical method allows analyzing carbon monoxide content only in gaseous media at excess air (oxygen) concentration. In the oxygen-depleted media, this method does not function. Interferential and optic methods are based on absorption of light by carbon monoxide, which allows determining a carbon monoxide concentration in its mixtures with an inert gas. The optic carbon monoxide determination method analyzes laser light reflected from beam-splitting mirrors; the probe beams are absorbed differently by carbon monoxide.

The abovementioned analysis methods require sampling and probes preparation for the analysis as well as complex equipment.

The present work is aimed at the study of the possibility to measure continuously the carbon monoxide and moisture concentrations in the CO + H₂O + N₂ gaseous mixture using a relatively simple in construction and operation high temperature solid electrolyte electrochemical sensor with the diffusion barrier.

Solid electrolytes are widely used to date in many fields of science and technology. Potentiometric and solid electrolyte sensors have proven to be efficient for the determination of oxygen, hydrogen and a number of other gases [1–11]. There is a number of successful works on the measurement of air humidity by the electrochemical cells with oxygen ion conductivity [12–15] as well as electrochemical cells based on the proton-conducting solid electrolytes [4,16,17]. The studies on the determination of the carbon monoxide and other combustion gases using solid electrolyte sensors are reported elsewhere [4,7,18–20].

The advantages of the electrochemical solid electrolyte sensors for the analytical measurements include their corrosion resistance in aggressive media, good reproducibility of the measurement results, simple operation and small dimensions.

Electrochemical properties of solid electrolytes and electrodes play a vital role in the operation of such electrochemical sensors. In the present paper we used a solid electrolyte with a unipolar oxygen conductivity based on the stabilized zirconium oxide of the 90 mol.% ZrO₂ + 10 mol.% Y₂O₃ composition; the electrodes were made of porous platinum. A diffusion barrier aimed at the gas-exchange process between the ambient media and the inner chamber atmosphere is an essential part of the sensor. Usually, a ceramic gas permeable layer of a solid electrolyte with a laser-made opening serves as a diffusion barrier [21–26]. The parameters of such diffusion barriers are hard to control and even harder to reproduce. At the same time, the sensor operating characteristics, including, the current values, depend on such parameters of the diffusion barrier as the channel diameter and length. In the studied sensor, we used ceramic capillaries made of alumina with 260 μm inner diameter and 20 mm length.

2. Experimental

2.1. Principles of the amperometric sensor operation for measurement of carbon monoxide and moisture in the gaseous mixture of the CO + H₂O + N₂ composition

To perform the present research, we developed a sensor composed of two isolated electrochemical cells: electrochemical cell 1 (EC1) and electrochemical cell 2 (EC2). Both cells have the same design and are made of the same material, i.e. oxygen-conducting solid electrolyte based on zirconium dioxide. EC1 is a carbon monoxide sensor that functions via the oxygen pumping from the analyzed gas flow to the inner sensor chamber. EC2 is a humidity sensor that functions via the oxygen pumping out of the sensor chamber into the flow of the analyzed gas mixture. Oxygen is extracted from the humid analyzed gas.

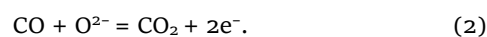
The sensor design is presented in Figure 1. Figure 2 demonstrates the sensor appearance. Solid electrolyte plates (Figure 1) have the size of 10x15 mm and the thickness of 1.5 mm. Both plates (2 and 10) have cavities on one side. The cavity depth is 0.9 mm. The intermediate solid electrolyte plate (6) does not have any cavities. On the opposite surfaces of the plates (2 and 10) platinum electrodes with platinum wire current leads are applied. Between the plates (2 and 6; 6 and 10) thin-wall ceramic capillaries (5 and 7) are located. Solid electrolyte plates and capillaries are joint with glass that serves as a good electric insulator between the cells. The sensor is located in a furnace that heats both the sensor and the analyzed gas to the desired temperature of 650~750 °C and maintains it using a Varta TP 703 controller (Varta Ltd., Russia) with the accuracy of ± 3 °C. The analyzed gas flow washes the sensor from outside and penetrates into both inner chambers (4 and 8) via the capillaries.

The voltage from DC sources CS-1 and CS-2 is supplied to the corresponding cells as shown in Figure 1. In the EC1 at the voltage of CS-1, an electrolytic decomposition of moisture at the outer electrode (1) results in the formation of oxygen, which is pumped into the inner chamber (4). In the EC2 oxygen is formed as a result of the humidity electrolysis when the voltage of the CS-2 is applied. The oxygen obtained in EC2 is pumped from the inner chamber (8) to the analyzed gas flow. In addition, the following reactions take place at the EC1 electrodes:

outer electrode



inner electrode



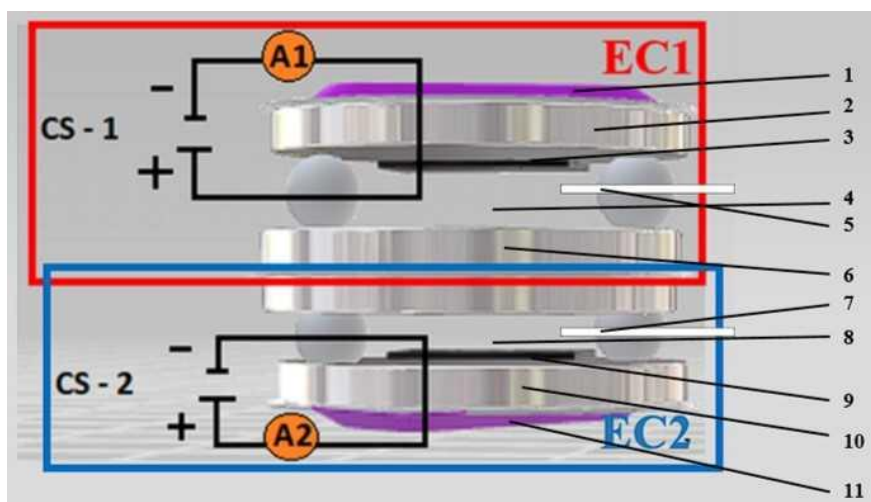


Figure 1 Schematic diagram of the electrochemical sensor: 1 – EC1 outer electrode for carbon monoxide determination, 2 – EC1 solid electrolyte plate for carbon monoxide determination, 3 – EC1 inner electrode, 4 – EC1 inner chamber for carbon monoxide determination, 5 – EC1 capillary for carbon monoxide determination, 6 – intermediate plate made of a solid electrolyte, 7 – EC2 capillary for moisture content determination, 8 – EC2 inner chamber for moisture determination, 9 – EC2 inner electrode for moisture determination, 10 – EC2 moisture solid electrolyte plate for moisture determination, 11 – EC2 outer electrode for moisture determination, A1 and A2 denote amperemeters, CS-1 and CS-2 denote sources of constant current.



Figure 2 Appearance of the YSZ-YSZ sensor with the intermediate layer and activated Pt electrodes.

The following reactions take place on the EC2 electrodes:

inner electrode



outer electrode



When operating with gas mixtures the oxygen pumping current into the EC1 for carbon monoxide determination reached 0.1 mA for CO - 1% and up to 3.5 mA for CO - 10%. The oxygen pumping off current of the EC2 inner chamber for moisture content determination was sufficiently lower and reached 0.1 mA for H₂O - 10%. As the applied voltage increased, the current grew, and at a certain voltage value the current stabilized at the limiting current value. The value of the limiting current is proportional to the carbon monoxide and moisture concentrations in the analyzed gas mixture. The value of the limiting current of the EC1 is expressed by the following equation [27].

$$I_{\text{lim}(\text{CO})} = \frac{2FD_{t(\text{CO})}SP}{RTL} X_{\text{CO}}, \quad (5)$$

where $D_{t(\text{CO})}$ is the CO diffusion coefficient in nitrogen; X_{CO} is the CO molar fraction in nitrogen; S is the capillary cross-section; P is the total pressure of the gas mixture; T is the

analysis temperature, K ; L is the capillary length; F is the Faraday number; R is the gas constant.

According to equation (5) the limiting current of the EC1 $I_{\text{lim}(\text{CO})}$ is directly proportional to the X_{CO} value and is determined by the carbon monoxide diffusion via the diffusion barrier. Similarly, the limiting current of the EC2 for the humidity control $I_{\text{lim}(\text{H}_2\text{O})}$ is directly proportional to the $X_{\text{H}_2\text{O}}$ value and it is determined by the moisture diffusion via the diffusion barrier. Thus, the limiting current for the moisture determination corresponds to equation [27]:

$$I_{\text{lim}(\text{H}_2\text{O})} = \frac{4FD_{t(\text{H}_2\text{O})}SP}{RTL} X_{\text{H}_2\text{O}} \quad (6)$$

To calculate the concentration of the determined component according to equations (5 and 6), the value of its diffusion coefficient, which depends on the temperature, is required:

$$D_t = D_0 \left(\frac{T}{273} \right)^\alpha \frac{1}{P}, \quad (7)$$

where D_0 is the standard value of the diffusion coefficient at 0 °C and D_t is the value of the diffusion coefficient at the desired temperature.

For the standard molecular diffusion, the limiting current of the cell is proportional to $T^{\alpha-1}$. For the gaseous mixture, where an inert gas (nitrogen, in particular) is the main component, the value of $\alpha-1$ is within the range of 0.5–0.8.

3. Results and Discussion

In the present work, we used cylinders with test gas mixtures of carbon monoxide-nitrogen, nitrogen, and a bubbler for gas mixture humidification. The humidity of gas mixture was controlled by a humidity sensor Moisture Monitor Series 3 (GESensingEMEA (Panametrics), Ireland).

Before the operation, the sensor positioned in the analyzed gas flow was heated up to the desired temperature using the furnace. The analyzed gas was supplied with the flow rate of 30 ml/min. Gas mixtures were supplied from the cylinders via reducers and mass-flow controllers F-201CV-100-RAD-AA-V (Bronkhorst High-Tech B.V., Netherlands) into the furnace with the sensor heated to the desired temperature.

It was mentioned that as the voltage applied on each electrochemical cell of the sensor increases, the current increases up to a certain value. Then as an increase in the applied voltage continues, the current stabilizes and the obtained current value is considered the limiting current. At the limiting current value and a specific temperature, one electrochemical cell determines the carbon monoxide concentration and another electrochemical cell determines the moisture concentration in the analyzed gas.

Figure 3 illustrates that the current of the EC1 increases and reaches its limiting value in the CO+3%H₂O+N₂ gas mixture. In addition, the limiting current appears for all three carbon monoxide concentrations in the potential region from 0.8 to 1.3 V. Figure 4 demonstrates changes in the EC2 currents caused by the applied voltage at the analysis of the gas mixture of the H₂O+3.7%CO+N₂ composition. In this case, the limiting current appears for all moisture concentrations ranging from 0.2 to 10% of H₂O at the voltage value of 0.7 V. It is seen that in the voltage region of 0.7–1.3 V for all analyzed moisture concentrations the limiting current is observed. Both for EC1 and for EC2 at the voltage above 1.3 V the current increases above the limiting value. This fact may be associated with the misbalance between the analyzed gas components (CO and H₂O) input via the diffusion barrier and pumped oxygen for EC1 and EC2. The appearance of the nonionic component of the zirconium dioxide-based electrolyte conductivity may be another reason for the current growth.

The formation of a stable signal and its correlation with the measurable parameter have a key role in operation of any sensor. In our case, when the sensor signal is obtained in the form of a limiting current, it should be interpreted for the determination of carbon monoxide and moisture concentrations. For the amperometric sensors the relationship between the measured values of the limiting current and the component concentrations are given by equations (5), (6) and (7). The main problem is a lack of reference data on the diffusion coefficients of these gases in the studied gas mixture for desired high temperatures. This fact complicates the calculation of the carbon monoxide and moisture concentrations in the studied gas mixtures at the set temperatures according to the values of the limiting currents.

Selection of an appropriate capillary is another problem to be solved for this type of sensors. Ceramic capillaries, used in the present work, have a small inhomogeneity in the diameter of the inner channel, which has a significant

influence on the value of the measured limiting current. Meanwhile, metallic capillaries may interact with the analyzed gas, and thus their usage is questionable. The deviation in the measured values may be caused only by the capillary, as the diameter of the ceramic capillary differs along the capillary length even though the capillaries are taken from the same batch. Therefore, the difference in the inner diameters of the capillaries does not play a decisive role in the application of amperometric sensors, because each sensor requires a preliminary calibration.

To eliminate the aforementioned factors, the sensors should be calibrated according to the obtained measurement results.

Figures 5 and 6 demonstrate the dependences of the limiting currents on the carbon monoxide and moisture concentrations in the temperature range from 650 to 750 °C. The obtained dependences are linear. The increase in the limiting currents associated with the temperature increase is explained by enhanced diffusion of the determined components according to equation (7).

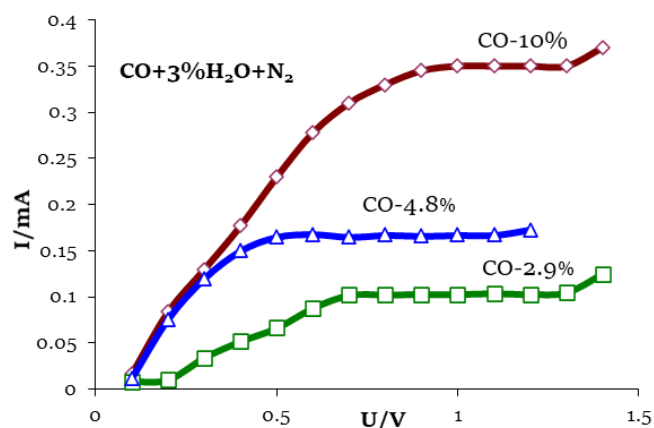


Figure 3 Current changes in EC1 for carbon monoxide determination on the applied voltage at 700 °C and the moisture content of 3%.

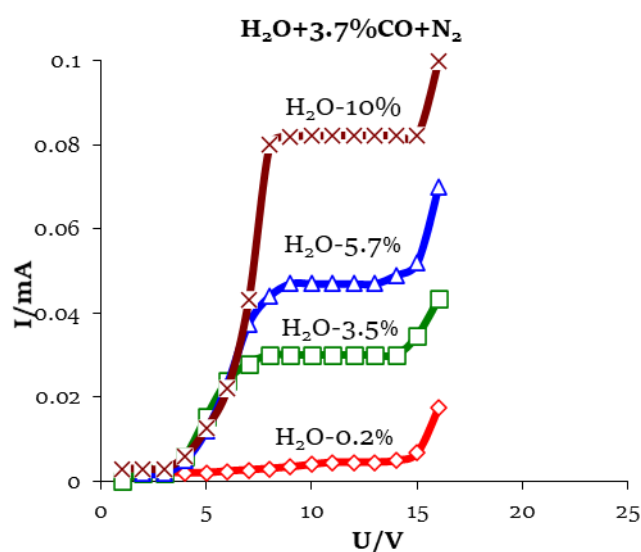


Figure 4 Dependences of the EC2 currents for measurement of the moisture on the applied voltage at 700°C and carbon monoxide concentration of 3.7%.

The dynamic characteristics of sensors for carbon monoxide and humidity measurements are presented in Figures 7 and 8.

The concentration of carbon monoxide in the gas mixture was measured in sequence of steps from 12.8% to 0.22% and back to 12.8%. The response of the sensor was simultaneously monitored for the change in the moisture concentration from 0.2 to 2.3%.

The sensor showed well reproducible results for the determination of both the carbon monoxide and humidity. In addition, the sensor response at the operation temperature of 700 °C is satisfactorily fast for the continuous measurements. Thus, the first response time considering the transport delay did not exceed 5–10 s, and the time required to reach 90% of the nominal concentration value of the measured component was about 1 min, considering the transport delay.

4. Limitations

Although the present study achieved promising results, there are still some limitations that should be acknowledged for future improvement of the sensor implementation. The YSZ-based solid electrolyte with oxygen conductivity provides satisfactory results, but to provide a wider applicability of the sensor it is desirable to decrease the operating temperature, which is a challenge. Therefore, new electrolyte materials with different types of conductivities and dopants are being tested to provide the desired result.

5. Conclusions

A solid electrolyte amperometric sensor with the diffusion barrier for rapid control of gaseous mixture composition during carbon monoxide conversion for hydrogen production has been studied. The sensor is composed of two electrochemical cells and allows simultaneous determination of carbon monoxide and moisture concentrations in the CO + H₂O + N₂ gas mixture. Electrochemical cells of the sensor are made of YSZ-based solid electrolyte with oxygen conductivity. In addition to the determination of the carbon monoxide concentration, the sensor operates by oxygen pumping into and out of the sensor chambers and, thus, allows determining the gas mixture humidity.

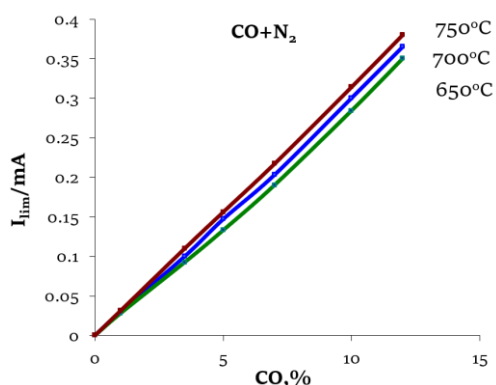


Figure 5 Dependences of EC1 limiting currents on the carbon monoxide concentration and temperature.

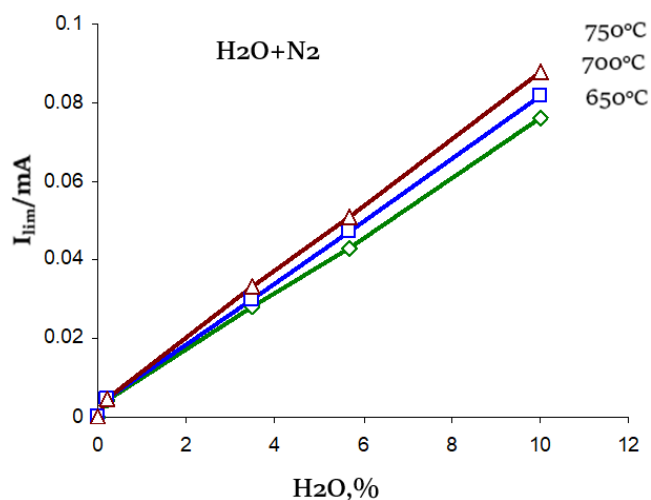


Figure 6 Dependence of the EC2 limiting currents on the moisture concentration and temperature.

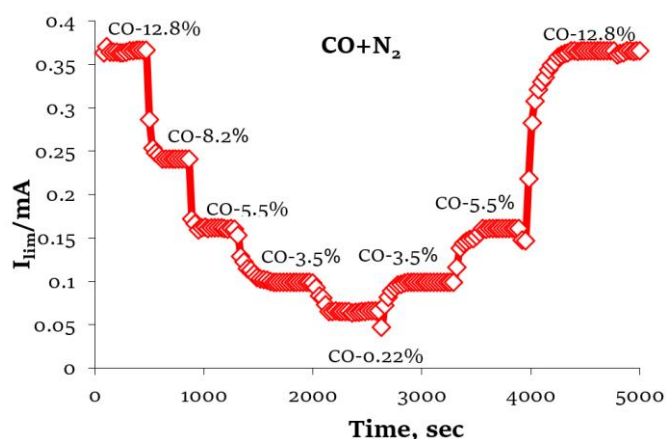


Figure 7 EC1 responses to the carbon monoxide concentration in the analyzed gas mixture at 700°C.

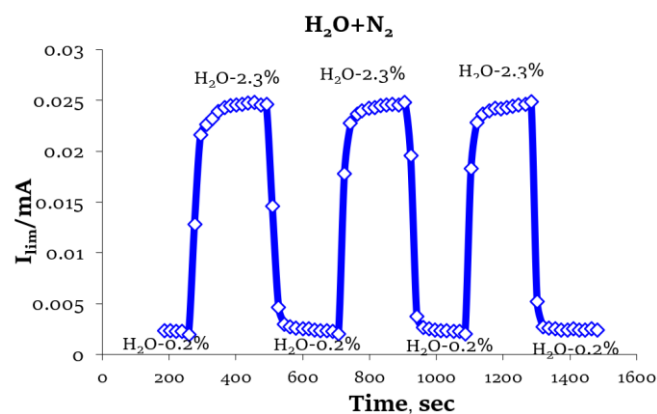


Figure 8 EC2 responses to the moisture concentration in the analyzed gas mixture at 700 °C.

The dependences of sensor currents on the applied voltage as well as dependences of the limiting currents on the carbon monoxide and moisture concentrations of the gas mixture were obtained for a number of temperatures. The temperature dependences of the limiting currents on the carbon monoxide and moisture concentrations in the gas mixture were determined in the temperature range of 650–750 °C. The dynamic possibilities of the sensor were detected.

The performed research elucidates a possibility to measure simultaneously carbon monoxide and moisture concentrations in gas mixtures using a solid electrolyte amperometric sensor and may become the basis for development of industrial scale sensors, for instance, for analysis of the initial gas flows during carbon monoxide conversion. This sensor is universal and may be used for the analysis of other binary gas mixture ($H_2O + H_2$; $CO+CO_2$ etc.), it is reliable and simple in production and operation, and it has miniature dimensions.

Supplementary materials

No supplementary materials are available.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Conceptualization: A.N.V., A.S.K.
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 Formal Analysis: A.N.V.
 Investigation: A.N.V., A.S.K.
 Methodology: A.N.V.
 Project administration: A.N.V.
 Resources: A.N.V.
 Supervision: A.N.V.
 Validation: A.N.V., A.S.K.
 Visualization: A.N.V.
 Writing – original draft: A.N.V.
 Writing – review & editing: A.N.V.

Conflict of interest

The authors declare no conflict of interest.

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