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ARTICLE TYPE

Se/C composite as cathode material for rechargeable lithium batteries with good electrochemical performanceLili Liu^a, Yuyang Hou^a, Yaqiong Yang^a, Minxia Li^a, Xiaowei Wang^a and Yuping Wu^{*a}

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A Se/C composite was prepared by a simple combination method of ball milling and low temperature treatment as a cathode material for Li/Se rechargeable batteries. It was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Its electrochemical performance as a cathode material for lithium rechargeable battery was tested by cyclic voltammetry (CV) and capacity measurements. Rate capacity and cycling performance of the as-prepared product are very satisfactory. Even at a current density of 500 mA g⁻¹, the composite can deliver a capacity of 187 mAh g⁻¹. The main reason is that the high conductivity of carbon decreases its charge transfer resistance and effectively suppresses the dissolution of oxidation products from the composite cathode.

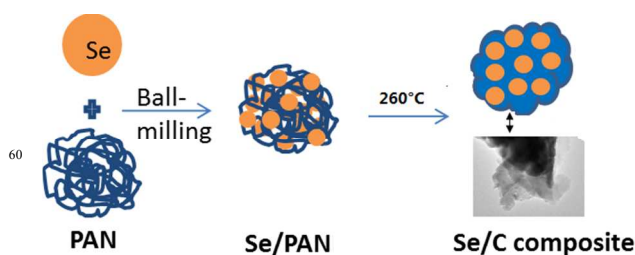
1. Introduction

The widespread application of mobile devices and potential use in electric vehicles has motivated scientific and technological efforts dedicated to improving existing rechargeable battery systems and to developing new electrode reactions and materials.¹ Currently, among all the known battery systems, lithium-sulfur batteries seem to have attracted considerable attention compared with the traditional lithium ion batteries because of their high theoretical capacity (1675 mA h g⁻¹) and energy density (2600 W h kg⁻¹).^{2,3} However, the poor electrical conductivity of S and the solubility of intermediary polysulfide species during cycling seriously impede their commercial applications.^{3,4}

In order to tackle the above restrictions, exploring novel electrode materials seems to be an imperative strategy.⁴⁻⁷ Selenium, owing to its similar chemical properties with sulfur, has been put forward as electrode material with great potential because of its higher electronic conductivity (approximately 20 orders of magnitude greater than that of S) and higher output voltages (at least 0.5 V higher for Li/S).^{4,5} Similar to Li-S battery, Li-Se battery is based on the reversible oxidization-reduction reaction between selenium and lithium. As a cathode material for rechargeable lithium battery, results from Amine's group⁴ indicate that although the charge potential can be up to 4.6 V (vs. Li⁺/Li) and the cycling performance is stable, it still suffers rather low columbic efficiency and capacity fading which are caused by the redox shuttle effect.⁸ Recently, new studies⁹ on the Se-based material show low voltage plateau during charge process which is much different from the other reports.^{4,5} Since many scientists have successfully tried using conducting materials such as carbon^{2c,10}, graphenes¹¹ and polypyrrole¹² to activate the electrochemical performance of the electrode materials because that these materials not only improve the electronic conductivity but also maintain the structural integrity of the electrode material. These results suggest that mixing with conductive material is a

good strategy for inhibiting the redox shuttle effect and thus improving the electrochemical performance of selenium electrode.

Here we report a Se/C composite prepared by a simple process as a cathode material for rechargeable lithium battery. The introduced carbon material effectively suppresses the dissolution of oxidation products of Se and greatly improves the electrochemical performance of the selenium cathode.

2. Experimental**2.1 Synthesis of Se/C composite**

Scheme 1 Schematic illustration for the preparation process of Se/C composite.

All the reagents were analytical grade. A certain amount of commercial Se particles and PAN (polyacrylonitrile) were loaded into a 50 ml steel bowl containing 18 hardened steel balls in a diameter of 10 mm. After high-energy ball-milling at a rotation rate of 400 rpm for 12 h on a planetary ball mill, the obtained uniform mixture was calcined at 260 °C for 12 h at a heating rate of 2 °C/min under flowing Ar (99.99%, 200 ml/min) to produce a Se/C composite with carbon in the internal and at the external surfaces of the Se nanoparticles. This simple preparation procedure is schematically illustrated in Scheme 1. The commercial Se particles, denoted as CP-Se, were used as a control.

2.2 Characterization and electrochemical evaluation

The following measurements were performed at room temperature. The crystal structures of the prepared Se/C composite and CP-Se were characterized by X-ray powder diffraction (XRD) using a Bruker Analytical X-ray System with CuK α radiation source filtered by a Ni thin plate. Scanning electron micrograph (SEM) and transmission electron micrograph (TEM) were obtained on a Philips XL30 scanning electron microscope and a JEOL JEM-2010 transmission electron microscope, respectively. Raman spectra was collected using a Raman spectrometer (JobinYvon HR800) employing a 10 mW helium/neon laser at 632.8 nm. X-ray photoelectric (XPS) spectra were obtained on a spectrometer (Perkin Elmer PHI-5000 C) with electron combination energy corrected by using graphite as a reference, whose combination energy of C_{1s} was designated at 284.60 eV.

Coin-type half-cells were assembled for evaluating the electrochemical performance of the as-prepared Se/C composite and the commercial Se. Commercial Se and the as-prepared Se/C nanocomposite were mixed with acetylene black (AB) and poly(vinylidene fluoride) (PVDF) at a weight ratio of 8:1:1 with N-methyl-pyrrolidone as a solvent. The mixture was coated on aluminium foil, then dried at room temperature and cut into small pellets with weight of about 2 mg for each pellet. Then the pellets were dried at 120 °C overnight in vacuum. Finally, button-type model cells were assembled in a glove box with the pellets as the working electrode, lithium foil as the counter and reference electrode, porous Celgard 2400 as the separator, and a standard 1.0 mol L⁻¹ LiPF₆ solution in a mixture of EC (ethylene carbonate) / DMC (dimethyl carbonate) / DEC (diethyl carbonate) (1:1:1, v/v/v) as the electrolyte. Electrochemical performance of both commercial Se and Se/C composite were carried out using a LANDct3.3 battery tester at different rates between 0.8 and 4.3 V vs. Li⁺/Li.

3. Results and Discussion

The composition and structure of the Se/C composite and CP-Se were characterized by X-ray diffraction (XRD) and Raman spectroscopy (see Fig. 1). In their XRD patterns (Fig. 1a), there are several peaks at 23.5° (100), 29.7° (101), 41.3° (110), 43.6° (102), 45.4° (111), 51.8° (201), 55.7° (112), and 61.5° (202), which are in good accordance with the diffraction peaks of the trigonal phase of selenium (JCPDS 06-0362).^{13,14} However, for the Se/C composite, no evident peaks for carbon or graphite can be observed. This suggests that the carbon exists mostly in amorphous state due to the heat-treatment at low temperature. For the Se/C composite, two characteristic bands are observed in the Raman spectrum of carbon: the one centred at 1343 cm⁻¹ (D band) is attributed to local defects and disorder area in the carbon, and the other at 1572 cm⁻¹ (G band) can be assigned to the sp² graphitized structure.^{11a} It is clear that the intensity of the D band is much stronger than that of the G-band, indicating that the carbon is mostly amorphous,^{11a,15} which is consistent with the above XRD patterns. However, the Raman spectrum of CP-Se reveals no characteristic bands of carbon. For the two materials, the resonance peaks at 233 cm⁻¹ are a characteristic of A₁ and E modes stretching of the trigonal selenium^{14,16} while the peaks at 460 cm⁻¹ can be attributed to the second-order spectra of the

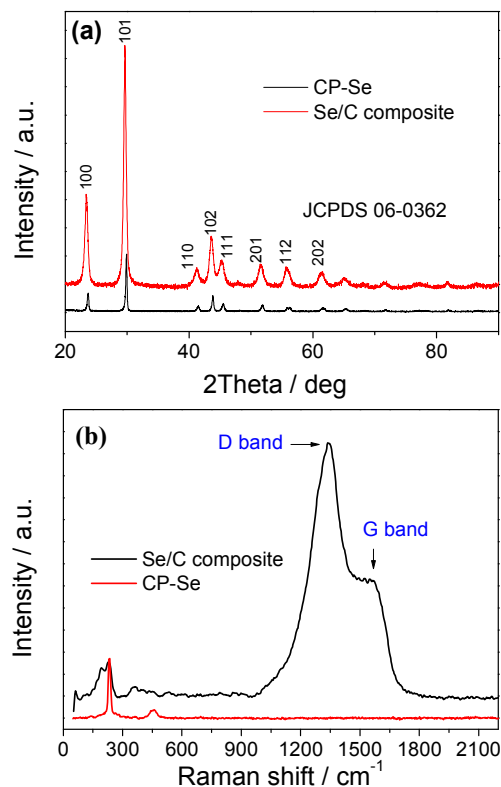


Fig. 1 (a) X-ray diffraction patterns and (b) Raman spectra of the Se/C composite and CP-Se.

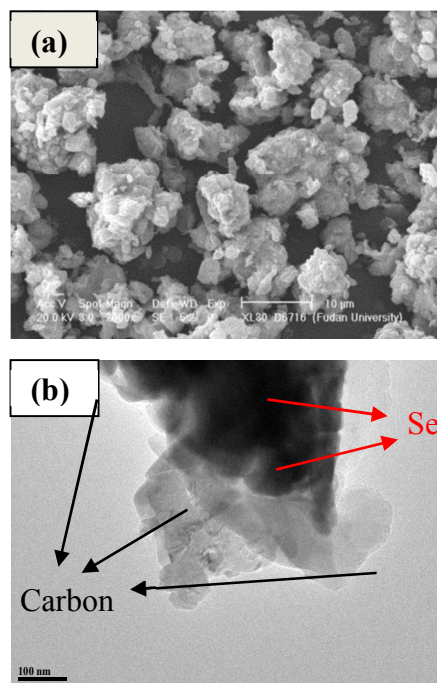


Fig. 2 (a) SEM and (b) TEM micrographs of the Se/C composite.

trigonal selenium.¹⁷

Fig. 2 illustrates the SEM and TEM micrographs of the Se/C composite. For comparison, the SEM micrograph of the CP-Se is shown in Fig. S1 in the ESI†. The Se/C composite shows morphology of particles with Se and C connecting with each

other closely, which can be proofed by the TEM micrograph (Fig. 2b). In Fig. 2a, it can be observed that the Se particle is embedded by the carbon material transformed from the PAN at 260 °C under Ar atmosphere. The EDX result shows that the weight ratio of the Se element in the composite is 56.4 %.

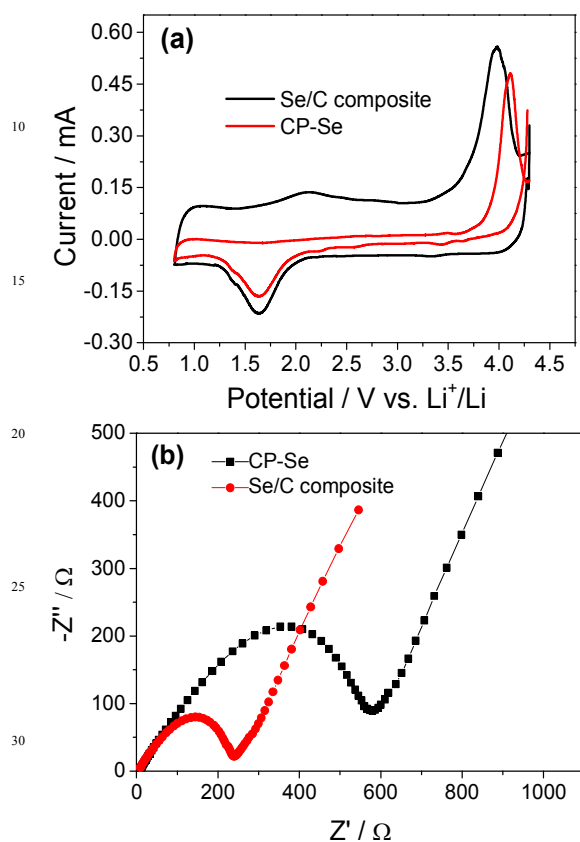


Fig. 3 (a) Cyclic voltammograms at the scan rate of 0.1 mV s^{-1} in the voltage range 0.8- 4.3 V and (b) Nyquist plots of the Se/C composite and CP-Se.

Cyclic voltammograms at the scan rate of 0.1 mV s^{-1} in the potential range 0.8- 4.3 V in the initial two scans of the as-prepared Se/C composite and the CP-Se are shown in Fig. 3a. It can be clearly observed that both electrodes show one pair of redox peaks: 1.6 / 4.1 V (vs. Li^+/Li) for the CP-Se while 1.6 / 3.9 V (vs. Li^+/Li) for the Se/C composite. In the first cycle, the peak located at 1.6 V corresponds to the Se reduction process and the peaks situated at 3.9 or 4.1 V belongs to the minimal structural changes to the electrode, not associated with an electrochemical reaction of the electrode materials.^{4,5} So, the CP-Se electrode only shows a single reduction peak at 1.6 V with the absence of no oxidation peak during the successive scanning process. Whereas, as for the Se/C composite electrode, an oxidation peak could be clearly seen in the CV plot at about 2.1 V, which can be ascribed to the Se oxidation. The above result well confirms that the Se/C electrode can partially activate the redox process and reduce the overpotential.^{12f}

Fig. 3b shows typical Nyquist plots of the Se/C composite and CP-Se. It is obvious that the charge transfer resistance (R_{ct}) relating to the electrochemical activities of the Se/C composite is significantly decreased compared with that of the CP-Se. This can be evidently ascribed the electronic conductive network of the

carbon from the decomposition of PAN at low temperature though it is amorphous.

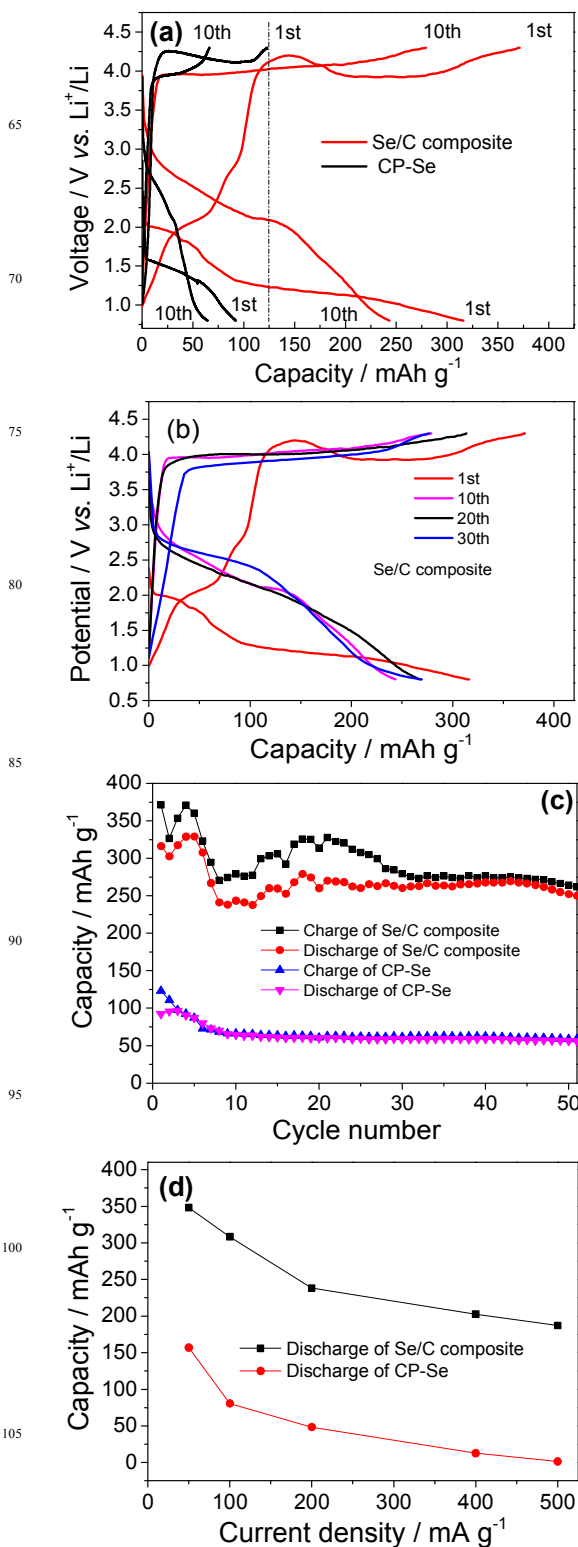


Fig. 4 (a) Discharge/charge curves in the first and 10th cycles of the CP-Se and the Se/C composite, (b) discharge/charge curves in the first, 10th, 20th and 30th cycles of the Se/C composite, (c) cycling performance at the current density of 100 mA g^{-1} and (d) rate capability at different current densities for the Se/C composite and the CP-Se in the voltage range between 0.8 and 4.3 V.

Fig. 4a displays the discharge/charge curves in the first and the 10th cycles of the Se/C composite and the CP-Se in the voltage range between 0.8 and 4.3 V at the current density of 100 mA g⁻¹. For both materials, the initial discharge involves one well-defined plateau indicative of the Li⁺ ion insertion (reaction of Li⁺ ions with Se via getting electrons). During the successive charge process, the prepared Se/C composite shows two plateaus (at 2.0 and 3.9 V) corresponding to the electrochemical reaction (conversion of Li₂Se into Se and Li⁺ ions via losing electrons) and the structural change of the Se,⁴ respectively. The charge and discharge phenomenon is similar with the reported data^{4,5} but different with the other studies.⁹ It is notable that the Coulombic efficiency in this work is much larger than that in the previous report.⁴ Unlike the Se/C composite, the CP-Se shows only one plateau. All the phenomena illustrated above are in good accordance with the above results from the CV test. Obviously, the Se/C composite can deliver a high capacity than that of the CP-Se electrode. After 10 cycles, the prepared composite can maintain a discharge capacity of 244 mAh g⁻¹, much larger than that of the CP-Se. There is no doubt that the addition of carbon in the composite creates a conducting network between the Se particles, resulting in a reduced contact resistance and an increase in the reaction area and capacity.^{2c,10,12} The voltage profiles at different cycle number are shown in Fig. 4b. It can be clearly observed that the difference between the charge and discharge voltages for the Li/Se-C cells is reduced with the increase of the cycle number, especially in the case of the voltage curves in the 30th cycle. Compared with the data presented by Amine's group,⁴ probably there are several factors accounting for the voltage difference. Firstly, electronic conductivity of carbon materials from the pyrolysis of polymers at low temperature (260°C) is not high as carbon nanotubes, which will cause large polarization. In addition, the as-prepared Se/C composite also present better cycling performance compared with the CP-Se. As shown in Fig. 4c, although the capacities in the first several cycles show a little fluctuation, they do not suffer drastic capacity fading. After 20 cycles, the Se/C composite can still maintain a discharge capacity of 269.5 mAh g⁻¹, but the CP-Se only delivers a discharge capacity below 60 mAh g⁻¹. From the 30 to 50 cycles, it presents a relatively stable charge and discharge capacity as well as higher coulombic efficiency, which is due to that the Se particle is embedded into the carbon material.

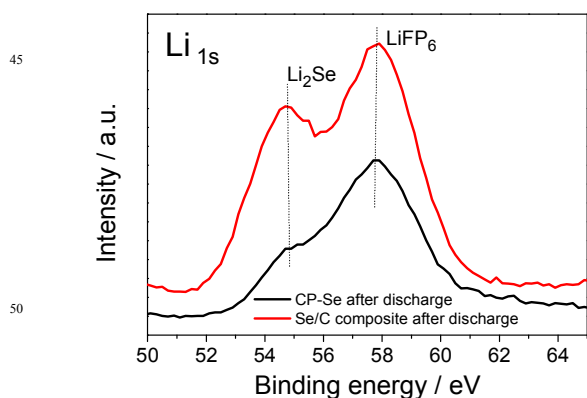


Fig. 5 XPS spectra of Li_{1s} in the Se/C composite and the CP-Se after the 5th discharge to 0.8 V.

To determine the rate performance of the as-prepared Se/C composite, the cathodes were tested galvanostatically at different current densities ranging from 50 mA g⁻¹ to 500 mA g⁻¹ (Fig. 4d). The Se/C composite exhibits obviously an improved electrochemical performance. A high discharge capacity of 348 mAh g⁻¹ was obtained at the current density of 50 mA g⁻¹, while that for the CP-Se is only 157 mAh g⁻¹. When the current density is increased to 500 mA g⁻¹, the Se/C composite can still keep a discharge capacity of 187 mAh g⁻¹. In contrast, the electrochemical performance of the CP-Se is poor. At 500 mA g⁻¹, its reversible capacity is almost 0. The better rate capability of the Se/C composite can be attributed to the improved conductivity of the introduced carbon matrix.

Fig. 5 shows the XPS spectra of Li_{1s} after the 5th discharge to 0.8 V. For both electrodes, the fitted curves clearly show an energy peak positioned at 54.7 eV, which corresponds to the component of the Li₂Se.^{12f} The peak at 58.6 eV may refer to the Li_{1s} of LiPF₆ from the electrolyte, which is not washed totally away. For the Se/C electrode, the peak at 54.7 eV is much stronger and sharper than that of CP-Se electrode, indicating a larger intensity of the Li₂Se in the electrode. This indicates that there is a deeper reaction between the Li⁺ ion and Se, which is the reason of higher capacity for the Se/C composite. At the same time, it also means a less quantity of Li₂Se dissolving into the electrolyte. All this can be attributed to the introduced carbon material closely on the inner structure and external surface of the Se particles. The above illustration is consistent with the result of Fig. S2 in the ESI†. After dismantling the cells of the two materials, the colour of the Celgard membrane for the Se/C cell is still white, while that for the CP-Se cell is red which is caused by the dissolution of the products such as Li_xSe, which is similar to dissolvable Li_xS.^{2a,18}

It is known that the electronic conductivity of carbon materials from the pyrolysis of polymers at low temperature (260°C) is not high. In this case, the carbon from the pyrolysis of PAN at such a low temperature can present good conductivity is due to its unique structure, which can cyclize at 200-250 °C to get a net-like polymers.¹⁹ When some small molecules are given off at this temperature, a graphene-like structure is achieved. Of course, its electronic conductivity is not very high. The main reason is that the temperature should not be too much above the melting point of Se (about 217 °C) in order to keep Se in the carbon. If carbon materials of higher conductivity are used to prepare a Se/C composite, its electrochemical performance will be better.

100 Conclusions

In summary, we have developed an Se/C composite by a very simple method as a cathode for rechargeable lithium battery with an excellent rate capability and good cycling performance. Even at a current density of 500 mA g⁻¹, the composite can deliver a discharge capacity of 187 mAh g⁻¹. The main reason is that due to the good electronic conductivity of carbon can increase the reaction depth and effectively suppress the dissolution of oxidation product from the cathode. The unique characteristics demonstrate that Se-based materials will be another promising cathode candidate for the future rechargeable lithium batteries.

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Notes and references

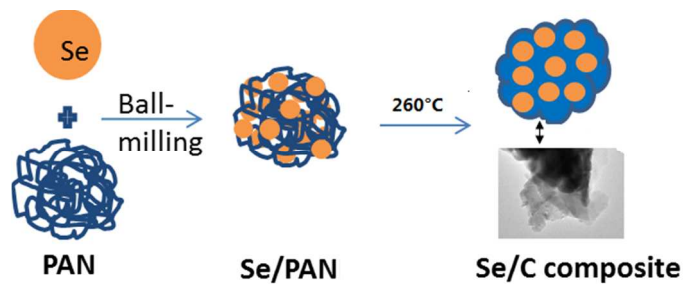
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- † Electronic Supplementary Information (ESI) available: SEM micrograph of commercial Se particles, and photos of the separator membranes after the 5th discharge to 0.8 V of the Se/C composite and the CP-Se electrodes. See DOI: 10.1039/b000000x/
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Table of content

: A Se/C composite as cathode material for rechargeable lithium batteries with good electrochemical performance

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¹¹ A Se/C composite is prepared as cathode material for rechargeable lithium batteries, which significantly enhances the capacity and improves the rate capability in comparison with the commercial Se particles.



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