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Influence of the Interlayer Thickness on the Mechanical Properties of Graphene Nanocomposites

Graphene nanocomposites (GNCs) have received significant attention due to their unique mechanical properties. In this study, we investigate the influence of the interlayer thickness on the mechanical performance of GNCs. The interlayer thickness was systematically varied, and the resulting mechanical properties were characterized using a combination of experimental and computational methods. The results highlight the importance of the interlayer thickness in tuning the mechanical properties of GNCs, which has implications for their potential applications in various fields.

Introduction

Graphene nanocomposites are a promising class of materials due to their exceptional mechanical properties. However, the interlayer thickness plays a crucial role in determining their performance. In this work, we explore the effects of varying the interlayer thickness on the mechanical properties of GNCs. The study provides valuable insights into the design of GNCs for specific applications.

Materials and Methods

The GNCs were prepared by exfoliating graphite and dispersing the resulting graphene nanosheets into a polymer matrix. The interlayer thickness was varied by controlling the exfoliation process. Mechanical tests were conducted using a universal testing machine, and the data were analyzed using statistical software.

Results

The results show that the interlayer thickness significantly affects the tensile strength and Young's modulus of the GNCs. As the interlayer thickness increases, the tensile strength decreases while the Young's modulus remains relatively constant. This trend is consistent across different types of polymers used in the study.

Conclusion

Our findings indicate that the interlayer thickness is a critical parameter in the design of GNCs for specific applications. Further research is needed to optimize the interlayer thickness for improved mechanical properties.

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Supporting Information

The supporting information includes detailed experimental procedures and additional results that are available upon request.

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spectrometer (ICP-MS)\textsuperscript{[9]} and an online electrochemical mass spectrometer (OLEMS) were used for real-time analysis of simultaneous electrode dissolution and product formation. This new approach brings us further in revealing the importance of electrode stability by showing electrode dissolution with unprecedented temporal and potential resolution and understanding the role of surface oxide formation on Pt during the reaction.

Results and Discussion

Comparison of different electrolytes

First, Pt dissolution was measured in methanol containing LiOH or NEt$_3$ during Kolbe electrolysis of acetic acid. In the course of neutralization of acetic acid with LiOH, one equivalent of water is released, which can significantly influence the reaction as well as the electrode dissolution behavior.\textsuperscript{[9c,10]}

Compared to this water-containing system, the system with NEt$_3$ base is practically water-free. Hence, an additional electrolyte system with 1 mol L$^{-1}$ LiOH, 0.1 mol L$^{-1}$ NEt$_3$ containing 0.1 mol L$^{-1}$ H$_2$O was also tested to pay heed to the effect of water. We conducted chronoaamperometric measurements in the EFC at three different anodic potentials: 1, 2, and 3 V [all potentials are referred to the ferrocene/ferrocenium (Fc/Fc$^+$) couple in this paper] in all three electrolytes. Substantial product formation in all three electrolytes was observed first at 3 V. Figure 1a shows the measurement results at an applied potential of 3 V for 3 min (for the other experiments, see the Supporting Information). The first two rows are the electrochemical data, and the subsequent rows show the simultaneously detected Pt dissolution and formed volatile products, respectively. The $m/z = 27$ value belongs to the Kolbe product C$_3$H$_6$ formed from acetic acid, while $m/z = 44$ represents CO$_2$ as a side product, and $m/z = 2$ corresponds to hydrogen evolution from methanol, water, or acetic acid reduction as the counter reaction. CO$_2$ could also stem from methanol oxidation, but as shown in the Supporting Information, we could unambiguously determine the source of CO$_2$ using $^{13}$C-isotopic labeled acetic acid. The reaction starts instantaneously as the anodic potential is applied. The somewhat later onset of the signals corresponding to C$_3$H$_6$ and CO$_2$ compared to H$_2$ is caused by the delayed permeation of the gases through the degasser membrane.

While quantification in terms of concentrations is not trivial, the Kolbe product can be detected in all three electrolytes. Although the current densities are somewhat similar in all three cases, the dissolution of Pt shows remarkable differences. Common in all three cases is the presence of both forms of dissolution.\textsuperscript{[9,10]} This observation seems to be independent of the Kolbe electrolysis since the same trend can be observed at the other two applied potentials, too (see the Supporting Information). The total dissolved amounts of Pt at different potentials to −0.5 V vs. Fc/Fc$^+$ for 10 min in methanol containing 1 mol L$^{-1}$ acetic acid and 0.1 mol L$^{-1}$ LiOH (left), NEt$_3$ (middle), and NEt$_3$ + 0.1 mol L$^{-1}$ H$_2$O (right), respectively. Applied potential (black), measured current density (gray), transient Pt dissolution (orange), H$_2$, $m/z = 2$ (green), C$_3$H$_6$, $m/z = 27$ (red), CO$_2$, $m/z = 44$ (blue).

Figure 1. (a) Kolbe electrolysis in the course of a chronoaamperometric measurement at 3 V vs. Fc/Fc$^+$ for 3 min followed by a potential step to −0.5 V vs. Fc/Fc$^+$ for 10 min in methanol containing 1 mol L$^{-1}$ acetic acid and 0.1 mol L$^{-1}$ LiOH (left), NEt$_3$ (middle), and NEt$_3$ + 0.1 mol L$^{-1}$ H$_2$O (right), respectively. Applied potential (black), measured current density (gray), transient Pt dissolution (orange), H$_2$, $m/z = 2$ (green), C$_3$H$_6$, $m/z = 27$ (red), CO$_2$, $m/z = 44$ (blue). (b) Total dissolved amounts of Pt at the potentials of 1 (blue), 2 (red), and 3 V (green) for a 3 min step followed by a 10 min at reductive potentials in LiOH (left), NEt$_3$ (middle), and NEt$_3$ + H$_2$O (right).

Pt dissolution in different electrolytes increases in the order of LiOH < NEt$_3$ ≤ NEt$_3$ + H$_2$O. Various reasons can be identified for that trend, and different factors have to be considered in the explanation, separately for the anodic and cathodic dissolution behavior. While anodic dissolution is associated with the direct dissolution of Pt in the form of Pt ions, the presence of a cathodic dissolution feature in the course of the potential step to −0.5 V is a sign for Pt−O formation at positive potentials.

The first striking observation is that although the LiOH-based system contains 0.1 mol L$^{-1}$ H$_2$O, the cathodic dissolution is very comparable to that in the NEt$_3$-based system, the water content of which is below 100 ppm. This phenomenon contradicts the common logic that higher water contents would cause
a higher grade of Pt oxidation, evoking a higher amount of cathodically dissolved Pt. However, there is a fundamental difference in the properties of the two cations found in the systems. Whereas Li\(^{+}\) ions are hydrophilic and keep water bound in their hydration shell, the hydrophobic NEt\(_3\)H\(^{+}\) cations solvate less water.\(^{[11,12]}\) The differences in the reactivity of hydrated and free water in nonaqueous media are described already in several publications.\(^{[11,12]}\) Therefore, even though more water is present in the LiOH-based electrolyte, it is less reactive at anodic potentials. Moreover, water molecules are transported away with Li\(^{+}\) ions from the positively charged electrode surface during the anodic potential step and cannot oxidize Pt to a much greater extent than the NEt\(_3\)-based water-free electrolyte.

The anodic dissolution profiles of the first two systems contradict the above explanation at first glance. In this case, the increased anodic dissolution in NEt\(_3\), despite the presence of more reactive water molecules, can be rationalized by considering the complexing effect of amines towards Pt.\(^{[15]}\) The effect is similar to that found in aqueous electrolytes containing halide anions.

Finally, it seems that for the water-containing NEt\(_3\) case, the anodic dissolution is only slightly lower but decreases faster, and cathodic dissolution increases, which leads to similar total dissolution values compared to the water-free system. Although the concentration of water is the same in both systems, the lower cathodic dissolution for the Li\(^{+}\)-containing system compared to the NEt\(_3\)-containing system results from the different hydration properties of Li\(^{+}\) and NEt\(_3\)H\(^{+}\) ions, as explained above. It is also worth mentioning that the dissolution increases in all electrolyte systems significantly from 1 V, at which potential only solvent oxidation occurs, to 2 V, at which potential Kolbe electrolysis commences. However, the dissolution does not increase significantly when 3 instead of 2 V is applied, even though more product formation is observed at higher potentials (see Figure 1b and the Supporting Information). This observation suggests that an increased radical concentration at the electrode surface has only a minor influence on the electrode dissolution rate. Unfortunately, the presented setup does not allow for the quantification of products and therefore the calculation of coulombic efficiencies (CE). The correlation of CE and electrode dissolution could be an important parameter for organic electrochemists to assess the economic feasibility of their reactions, but this would require an even more complex setup, which is the subject of further developments and investigations.

### Influence of electrolysis time

Even though the dissolution rate is in the order of some 100 pg s\(^{-1}\) cm\(^{-2}\) during the electrolysis, which might seem to be negligibly low, in long-term electrolysis, the direct material loss can also accumulate to a more substantial value using electrodes of some 100 cm\(^2\) size. This could cause severe performance degradation, counter electrode modification, and product contamination. To estimate the dissolved amount, we have conducted chronoamperometric (CA) measurements at a constant anodic potential for different times in the water-containing and water-free NEt\(_3\)-based electrolytes. The results are shown in Figure 2. The anodic dissolution in the water-containing system is somewhat suppressed compared to the water-free system and converges in both cases to a stable value after a transient signal. This feature is especially well observable as the electrolysis time increases. Therefore, to be able to estimate the total dissolved amount after longer electrolysis times, the rate of the anodic dissolution curve after the transient signal gives a more realistic estimation than a simple linear fit of data points in Figure 2c (see the Supporting Information). Especially since longer electrolysis times are more realistic in real applications, the contribution of initial transient dissolution diminishes. The stable value lies at around 90 pg s\(^{-1}\) cm\(^{-2}\) in the water-free system. That means in the case of electrolysis over one day using an electrode with a surface area of 1 m\(^2\) results in the dissolution of 77.8 mg Pt. Over one year, this accumulates to about 28.4 g, which is 13.3\% of the mass of a 10 \(\mu\)m thick electrode. From the results, we can also see that water has a beneficial effect on Pt dissolution. In the water-containing electrolyte, the material loss accumulates to 8.1\% in the above example. As mentioned in the introduction, aside from the material loss, secondary effects might interfere with the desired reaction. Considering a lab-scale electrolysis in a 3 mL solution using a Pt electrode with 1 cm\(^2\) electrode area, the 90 pg s\(^{-1}\) cm\(^{-2}\) Pt dissolution rate in the anhydrous electrolyte would result in an unknown and unwanted homogenous catalytic effect.

The dissolution curves corresponding to the series of electrolysis steps carried out for different duration show that the extent of cathodic dissolution is independent of the length of time spent at the anodic potential. In addition, this phenomenon is independent of the presence of water in low or high concentration in the system. This observation completely contradicts the behavior of aqueous systems, in which the extent of cathodic dissolution strongly depends on the time spent at anodic potentials, where oxide and subsurface oxide formation take place.\(^{[14]}\) The occurrence of oxide formation is evidenced by the cathodic dissolution; however, its independency from the electrolysis time suggests that the surface is protected from extensive oxide formation during the reaction.

### Electrode dissolution during cyclic voltammetry

In order to go deeper into the mechanistic details of the processes, we have carried out potentiodynamic measurements with the abovementioned three electrolyte systems and isotopically labeled acetic acid (see also the Supporting Information). Figure 3 shows cyclic voltammetry (CV) measurements together with the simultaneously detected dissolution and product formation curves as a function of time. The three columns represent the situations with the different bases added to acetic acid in the methanol-based electrolyte. The potential was cycled between −0.5 and 3 V with a scan rate of 10 mVs\(^{-1}\).
In systems with 0.1 mol L\(^{-1}\) water present (LiOH and NEt\(_3\) + H\(_2\)O), the onset of oxidation current is shifted to more negative potentials by 300 mV. As obvious from the mass spectrometric signals of C\(_2\)H\(_6\) and CO\(_2\), this first oxidation process does not correspond to the Kolbe electrolysis but is characteristic for the oxidation of the solvent methanol. The shift in the onset of the methanol oxidation reaction (MOR) could be explained by the increasing availability of OH\(^-\) species at the surface, which are known to be essential for methanol oxidation in aqueous media.\(^{[13]}\) Note that the methanol oxidation at such low water concentration does not result in the formation of CO\(_2\) even at higher potentials, as evidenced from measurements with isotopic labeled acetic acid (see Figure S5).

Pt dissolution for the system containing NEt\(_3\) starts at around 0.8 V, approximately 400 mV more negative than for the system containing LiOH. Since NEt\(_3\) is a weaker base (p\(K_B\) = 2.99) than LiOH (p\(K_B\) = 0.18), both species NEt\(_3\)H\(^+\) and NEt\(_3\) exist. The complexion properties of NEt\(_3\) could be the reason for the increased Pt dissolution at lower potentials.\(^{[13]}\) The addition of water to the NEt\(_3\) system does not limit the anodic Pt dissolution significantly, probably because of the stronger Pt–NEt\(_3\) interaction compared to Pt–H\(_2\)O interaction. However, the expected increased cathodic Pt dissolution associated with the reduction of Pt–O can be observed. The onset of the oxide reduction, which is expressed as cathodic dissolution in the analysis, is shifted around 600 mV to negative potentials compared to pure methanol.\(^{[16]}\) We speculate that the substituted oxide layer, as described by Conway and Vijh,\(^{[16]}\) is the

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**Figure 2.** Kolbe electrolysis in the course of chronoamperometric measurements at 3 V vs. Fc/Fc\(^-\) for varying anodic polarization times (10, 5, 3, 1 min) followed by a potential step to −0.5 V vs. Fc/Fc\(^-\) for 10 min in methanol containing 1 mol L\(^{-1}\) acetic acid and (a) NEt\(_3\) and (b) NEt\(_3\) + 0.1 mol L\(^{-1}\) H\(_2\)O, respectively. Applied potential (black), measured current density (gray), transient Pt dissolution (orange), H\(_2\): m/z = 2 (green), CH\(_2\)CO\(_2\): m/z = 27 (red), CO\(_2\): m/z = 44 (blue). (c) Total dissolved amounts of Pt as the function of the duration of the anodic potential step at 3 V.
reason for this shift. The strongly adsorbed CH$_3$COO species stabilize the underlying oxide. Only after their removal by the negative potential excursion, the reduction of oxide becomes possible. This mechanistic observation is summarized in the Table of Contents graphic.

The method was also applied during galvanostatic measurements at 5, 10, 15, and 20 mA cm$^{-2}$ in methanol containing 1 mol L$^{-1}$ acetic acid and 0.1 mol L$^{-1}$ LiOH to illustrate the electrode dissolution behavior under galvanostatic conditions. For details, we refer to the Supporting Information.

Conclusions

We have described the simultaneous, time- and potential-resolved electrode dissolution and gaseous product monitoring with a high resolution during the Kolbe synthesis reaction. We have shown the crucial effect of base and water addition on the stability of the electrodes. Furthermore, with the help of our approach, we unveiled hitherto unknown features of the reaction, especially with regard to dissolution mechanisms. We have demonstrated that the detected amounts of dissolved platinum are significant and might affect the technical implementation of the electrolysiss reaction. With that, we want to emphasize the importance of such investigations for similar reactions in the future, utilizing other, in some cases, more reactive metals with respect to dissolution. Even at relatively high water concentrations, Pt is protected from the continuous formation of oxide by an adsorbed layer. This observation is evidenced by the cathodic dissolution independent of the time of previous anodic polarization. It is also very possible that this same adsorbed layer prohibits the reduction of Pt–O that is reflected in a highly shifted onset of cathodic dissolution during cyclic voltammetry measurements.

Experimental Section

Chemicals and materials

The electrolytes were prepared from methanol (max. water content 0.003 %, SeccoSolv, Merck), acetic acid ($\geq$ 99 %, HiPerSolv CHROMA-NORM, VWR Chemicals), anhydrous LiOH (98 %, Alfa Aesar), NEt$_3$ (99 %, Alfa Aesar), and ultrapure water (18 M$\Omega$ cm) from a Milli-Q IQ 7000 system (Merck). All chemicals were used as received without further purification. The water content of the used electrolytes was measured using a Karl Fischer 917 Coulometer (Metrohm). All electrolytes were prepared and the water content was determined in an Ar-filled glovebox (MBraun), where H$_2$O and O$_2$ contents were kept below 0.1 ppm. Determined water contents of electrolytes: methanol containing 1 mol L$^{-1}$ acetic acid and 0.1 mol L$^{-1}$ LiOH: 2275 ppm, NEt$_3$: 65 ppm, NEt$_3$ and 0.1 mol L$^{-1}$ H$_2$O: 2305 ppm.

Measurement setup

The details of the homemade EFC coupled with a Nexion 2000 ICP-MS (PerkinElmer) setup for on-line electrode stability investigations in nonaqueous media were previously described.[9a] For the on-line stability investigation of electrode materials during electrosynthesis, a homemade degasser was added between the outlet of the EFC and the ICP-MS for product formation control (see Figure 4). The degasser consisted of a Silastic membrane mounted in a Swagelok T-piece with two short fused silica pieces at the inlet and outlet of the T-piece inserted into the membrane tube and short pieces of flexible tubing connecting the degasser with the outlet of the EFC.
and the inlet of the ICP-MS. The stainless-steel capillary (Vici Jour) that connected the degasser with the MS was 1.2 m long and had an inner diameter of 0.18 mm. The capillary was heated to 150 °C to prevent condensation of products in the capillary. The size of the capillary was optimized to the pumping rate of the turbomolecular pump of the MS. The measured capillary vacuum was \( p_{\text{ev}} = 7 \times 10^{-11} \) mbar, and the vacuum in the analyzer chamber was \( p_{\text{in}} = 5 \times 10^{-10} \) mbar.

The electrolyte was pumped through the EFC with a flow rate of 150 \( \mu \text{L min}^{-1} \) using a syringe pump (Legato® 100, KD Scientific Inc.). The working electrode, polycrystalline Pt (99.99 %, MaTeK), was polished prior to measurements using 0.3 \( \mu \text{m Al}_2\text{O}_3 \) paste on a MD-Mol polishing cloth (Struers). A glassy carbon rod (HTW Hochtemperatur-Werkstoffe GmbH) was used as a counter electrode (CE) and positioned upstream of the working electrode to prevent Pt ion reposition on the CE. As reference electrode (RE), a homemade leakage-free RE (cell body purchased from Innovative Instruments, Inc.) was prepared by immersing an Ag wire in methanol containing 0.1 molL\(^{-1}\) tetrabutylammonium perchlorate and 0.01 molL\(^{-1}\) AgNO\(_3\).

**Electrochemical measurements**

All electrochemical measurements were conducted using a VSP 300 potentiostat (BioLogic). Prior to measurements in the EFC, the homemade reference electrode was calibrated against the \( \text{Fc/Fc}^+ \) couple in a glass cell using the same electrode materials and electrolytes. Before the measurements, the internal resistance in the EFC was determined with electrochemical impedance measurements. Resistance varied between 40 and 200 \( \Omega \) depending on the conductivity of the electrolyte system and the exact electrode position. 85 % of the determined internal resistance was compensated for during the measurement using the manual internal compensation mode of the potentiostat. No post-measurement cleaning cycles were performed with a scan rate of 200 mV s\(^{-1}\) between −0.5 and 3 V.

**ICP-MS measurement conditions**

ICP-MS measurement conditions for methanol-based electrolytes were previously described in detail.\(^{[4]}\) For calibration of the ICP-MS, calibration standards in the electrolyte matrix with concentrations of 0.5, 1, and 5 \( \mu \text{g L}^{-1} \) were prepared from \( \text{H}_3\text{PtCl}_6 \) in HCl 7 % (Certipur, Merck). The internal standard (10 \( \mu \text{g L}^{-1} \) Re in ethanol) was prepared from (NH\(_4\))\text{ReO}_4 in water (Certipur, Merck).

**OLEMS**

A QMG 700 quadrupole mass spectrometer (Q-MS) (Pfeiffer Vacuum, Asslar) was equipped with an electron ionization (EI) ion source (ionization voltage set to 70 V) and a secondary electron multiplier (SEM) detector for the gas evolution measurement.

**Synchronization of measurements and delay time**

To determine the delay time and synchronize the electrochemical measurement with the ICP-MS and Q-MS data before each measurement, a potential of 1.5 V was applied for 10 s. At this potential, Pt dissolution and \( \text{H}_2 \) evolution occurred. The onset of Pt dissolution and \( \text{H}_2 \) evolution was determined as the time at which the intensity of the ICP-MS and Q-MS signals exceeded three times the standard deviation of the background signal. The delay time is the difference between the times of the onsets and the beginning of the potential pulse. The delay times are 60 s for the ICP-MS and 35 s for the Q-MS.

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**Conflict of Interest**

The authors declare no conflict of interest.

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