Mechanoelectronics: Flexible Organic Semiconducting Single Crystals for Durable All-Flexible Field–Effect Transistors


Abstract: Although many examples of mechanically flexible crystals are currently known, their utility in all-flexible devices is not yet demonstrated, despite their immense potential for fabricating high performance flexible devices. We report two alkylated diketopyrrolopyrrole (DPP) semiconducting single crystals, one of which displays impressive elastic mechanical flexibility whilst the other is brittle. Using the single crystal structures and density functional theory (DFT) calculations, we show that the methylated diketopyrrolopyrrole (DPP-diMe) crystals, with dominant π-stacking interactions and large contributions from dispersive interactions, are superior in terms of their stress tolerance and field-effect mobility (μEET) when compared to the brittle crystals of ethylated diketopyrrolopyrrole derivative (DPP-diEt). The field effect transistors (FETs) made of flexible substrates using elastic microcrystals of DPP-diMe retained μEET (from 0.019 cm²/Vs to 0.014 cm²/Vs) more efficiently even after 40 bending cycles when compared to the brittle microcrystals of DPP-diEt which showed a significant drop in μEET just after 10 bending cycles. Our results not only provide valuable insights into bending mechanism, but also demonstrate the untapped potential of mechanically flexible semiconducting crystals for designing all flexible durable devices.

Introduction

Organic crystalline materials, which enable many applications in the fields of electronics,1–3 smart actuators,4 sensors,5,6 etc., have gained tremendous recent attention in material science due to their unique advantages like room temperature processability, tunability, low cost, etc.7 Although high crystallinity is critical for obtaining optimum device performance,7,8 this often comes with a counter effect, i.e., increased mechanical brittleness,8,9 severely hampering the durability of the devices. Contrary to the general perception that all organic crystalline materials are brittle, many recent studies have demonstrated that a small subset of organic crystals exhibit exceptional mechanical flexibility with practical relevance.9,12

Systematic studies have shown that the mechanical flexibility or stress tolerance ability of molecular crystals strongly depends on the nature of intermolecular interactions and packing of molecules in crystals.3–9,12 Although significant progress has been made in understanding of structure-mechanical property correlations in the field of crystal engineering, controlling mechanical flexibility in crystalline materials still remains an extraordinary challenge. Recent reports on several organic single crystals with exceptional elastic flexibility have shown their attractive applications in wave-guides,5,6,12 ferroelectrics17,26 and semiconducting devices.8,18,27 However, to the best of our knowledge, the utility of the mechanically flexible single crystals in all-flexible devices is yet to be demonstrated. For instance, Gong and co-workers recently found a unique way to grow single crystals of coronene in bent condition and used different device geometries for demonstrating phototransistor properties and other applications.26 However, their devices do not involve any “mechanical flexing” as the substrates used were rigid.

Utilizing the recent critical insights gained from the understanding of elastic molecular crystals,9,11,17,19,20,25,28,29 here we demonstrate the advantage of elasticity in semiconducting single crystals for fabricating mechanically flexible and durable electronic devices, as compared to structurally close brittle single crystals. Hence, our correlation study, connecting molecular and supramolecular level information to the mechanical flexibility in the studied semiconducting single crystals, has implications for the design of mechanically durable and efficient crystalline organic electronics for use, in flexible field-effect transistors (FET) and other optoelectronic applications, for example via a bottom-up approach (molecule-to-crystal-to-device) using crystal engineering principles. There are reports of field-effect transistors (FET) with high mobility values than what we obtained over here but it is to be noted that our values are comparable to the other reported FETs fabricated using flexible substrates.30 Nevertheless, our aim in this study is to showcase the proof of concept of the importance of fabricating flexible semiconducting crystals to develop durable all flexible FET than a brittle one.

Results and Discussion

As a proof of concept, we prepared two derivatives of a well-known p-type semiconductor molecules, diketopyrrolopyrrole (DPP), substituted with alkyl groups, namely dimethyldiketopyrrolopyrrole (DPP-diMe) and diethyl diketopyrrolopyrrole (DPP-diEt) (Figure 1a, b). We introduced the simple alkyl groups based on the design strategies reported for soft-crystals.8,9,31 Aromatic systems with such functional groups typically close pack via π-stacking and other dispersive interactions in crystal structures which tend to accommodate local molecular movements under stress, thus in general imparting mechanical softness to the crystals.8,31 Acicular single crystals of DPP-diMe were obtained from
dichloromethane and toluene solution, whereas single crystals of DPP-diEt were obtained from toluene solution by the slow evaporation method in 5-7 days, which were amenable for all further studies.

Results from qualitative mechanical bending tests\textsuperscript{14} showed that crystals of DPP-diMe bend smoothly (Figure 1c) into a loop. Upon release of external mechanical stress, the crystals recovered to their original shape without any sign of permanent deformation.

Figure 1. Molecular structure (inset) and optical images of single crystals of (a) DPP-diMe and (b) DPP-diEt. Micrographs showing the bending steps in single crystals of (c) DPP-diMe (elastic) and (d) DPP-diEt (brittle), during the qualitative three-point flexural mechanical tests.

Figure 2. Crystal packing of DPP-diMe crystals. (a) π-Stacked columns, extended along crystal growth direction, with a slip-stack angle of 45^\circ and 3.298 Å interplanar distance and (b) corresponding pairwise total interaction energy, \( E_{\text{total}} = -69 \text{ kJ mol}^{-1} \) (calculated from the CIF obtained from SCXRD). (c) and (d) Packing patterns viewed along different directions.

\( \text{Figure 1.} \) Molar ature (inset) and optical images of single crystals of (a) DPP-diMe and (b) DPP-diEt. Micrographs showing the bending steps in single crystals of (c) DPP-diMe (elastic) and (d) DPP-diEt (brittle), during the qualitative three-point flexural mechanical tests.

Confirming their elastic nature (see Supplementary Movies S1, S2), testing the mechanical properties of DPP-diEt crystals of comparable morphologies under similar conditions, led to no appreciable elastic flexibility. Instead, crystals of DPP-diEt fractured in a brittle manner (Figure 1d, Supplementary Movies S1, S2), a property which is characteristic to most molecular crystals. The maximum strain \( \varepsilon \) that the elastic crystal (length: 2 mm; thickness: 0.0101 mm) of DPP-diMe could withstand during mechanical bending was in the range of 3 – 6 \% (for details see the Supporting Information, Figure S1). On the other hand, the acicular crystals of DPP-diEt fractured without any noticeable bending deflection, confirming their poor strain tolerance as compared to the DPP-diMe crystals. To rationalize the difference in the macroscopic mechanical behaviour, we investigated the two crystals using single crystal X-ray diffraction (SCXRD), nanoindentation, energy frameworks (using CrystalExplorer 17.5) and density functional theory (DFT) methods. DPP-diMe crystallizes in the monoclinic space group \( \text{P}2_1/\text{n} \) with half a molecule in the asymmetric unit. Molecules with nearly planar conformation are π-stacked along the \( a \)-axis (crystal growth axis) to form parallel columns in the structure (Figure 2a-c). The interplanar distance of stacks is 3.298 Å with a slipping angle of 45º (Figure 2a). Molecules of adjacent stacked columns are mutually oriented such that the methyl groups of one column point to the thiophene ring of another column (Figure 2d). π-Stacking is a dominant feature of the structure with only weak \( \text{sp}^2 \)C–H···O and other dispersive contacts in the structure. The energy frameworks calculations further confirm that the π-stacking \( (E_{\text{total}} = -69 \text{ kJ mol}^{-1}, \text{see Supporting Figures S2, S3}) \) interactions dominate the packing (Figure 2b). Contributions from other dispersive interactions are significantly smaller with

![Figure 2](image-url)
$E_{\text{total}} = -17 \text{ kJ mol}^{-1}$ and $-15 \text{ kJ mol}^{-1}$ (see Supporting Information Figures S2, S3). The orientation of columnar packing with respect to both major (011) and minor (01-1) faces is comparable (Figure 1c). The nearly corrugated arrangement of molecules makes the crystal structure devoid of any flat slip planes despite the presence of dispersive interactions in orthogonal directions, i.e., strong π-stacking and relatively weak (sp$_2$)C–H···O and other dispersive interactions.

Presence of dispersive interactions with large energy contributions and planar conformation of molecules, and absence of facile slip planes, seem to favour the appreciable elastic flexibility in crystals of DPP-diMe.$^{8,9,11,29}$ Upon mechanical bending of the crystals on the major face (011), molecules are expected to experience local changes to accommodate an expansion of the outer arc and contraction of the inner arc (Figure 3a). Upon the release of stress, the molecules regain their original positions, allowing the crystal to restore its original macroscopic shape. Generally, crystals with strong three-dimensional networks and those with rigid structures (specific interactions) deform in a brittle manner.$^{8,13,31}$ Crystals dominated by large dispersive interactions, particularly the parallel columns with dominant π-stacking, have been shown to allow reversible local molecular movements, thus making them exceptionally elastic.$^{11,16,20,25,28,29}$ Crystals with facile slip planes may show a small elastic response but eventually deform plastically, limiting their restoration ability. In the case of DPP-diMe, we did not observe any plastic deformation during bending in our experiments.

The DPP-diEt also crystallizes in the monoclinic space group P2$_1$1/n with half a molecule in the asymmetric unit. However, here the molecule is non-planar with a “Z-shaped” conformation as the alkyl substituents with an extra –(CH$_2$)$_2$– group are oriented nearly perpendicular to the molecular plane (Figure 3a). Similar to DPP-diMe, these crystals also stack along the a-axis via π-stacking with an interplanar distance of 3.373 Å and slipping angle of 40° (Figure 3b). The (011) and (01-1) faces were found to be the major and minor faces, respectively. The π-stacking interactions are slightly weaker ($E_{\text{total}} = -61 \text{ kJ mol}^{-1}$) here. There are also other dispersive interactions in the structure with $E_{\text{total}} = -18 \text{ kJ mol}^{-1}, -11 \text{ kJ mol}^{-1}$ and $-19 \text{ kJ mol}^{-1}$. In $E_{\text{total}} = -19 \text{ kJ mol}^{-1}$, the major contribution is (sp$_2$)C–H···O having a maximum $E_{\text{ele}}$ component of $-12.4 \text{ kJ mol}^{-1}$ (see the supporting information S4, S5). In the structure, four DPP-diEt molecules form a tetramer through the moderately strong (sp$_2$)C–H···O ($d^\prime$ Å; $\theta^\prime$: 2.49Å, 164.07°) hydrogen bonding interactions (Figure 3d). The electrostatic potential surfaces show that the carbonyl oxygen of DPP-diEt is more polarizing than that in DPP-diMe, which is consistent with the observation of stronger (sp$_2$)C–H···O hydrogen bonding interactions in the former (Figure S6). We have not observed slip parallel to the needle axis in these crystals, which suggests that the activation energy for the sliding of layers is higher than that of the fracture energy.$^{8,9,14}$ Our analysis reveals

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**Figure 3.** Crystal packing of DPP-diEt with the 3D topology of energy frameworks. (a) “Z-shaped” DPP-diEt molecule. (b) π-Stacked molecules with an interplanar distance of 3.373 Å and slip-stack angle of 40°. (c) Energy frameworks showing pairwise total intermolecular interaction energies (calculated from the CIF obtained from SCXRD). The thickness of the tube showcases the magnitude of the corresponding pairwise energy. (d) View of the hydrogen-bonded 2D sheet of DPP-diEt in ab plane; (e) Packing pattern showing the indentation direction.
that the interactions perpendicular to the stacking direction are relatively stronger and more specific in DPP-diEt as compared in DPP-diMe. Moreover, the non-planarity of the DPP-diEt molecule also might be contributing to the rigidity of the stacks, preventing local movement (rotation or splaying of stacked molecules). Short range and long-range molecular movements form the basis for elastic (reversible) and plastic (irreversible) bending, respectively, in molecular crystals. Hence, the rigid structure of DPP-diEt can be attributed to the observed brittleness in its crystals upon stressing on (011)/(01-1).

Further, we quantified the mechanical properties of the two crystals by performing nanoindentation tests on the (011) major faces of DPP-diMe and DPP-diEt crystals at a peak load of 6 mN (Supporting Figures S7, S8). The typical load (P) – depth (h) curves obtained from the nanoindentation on two crystals are given in Figure 4a. The Young’s modulus (E) and nanohardness (H) for DPP-diMe crystals are 6.47 ± 0.16 GPa and 0.24 ± 0.01 GPa, respectively (Figure 4a). However, in the case of DPP-diEt single crystal, the E and H are slightly higher, 8.64 ± 1.09 GPa and 0.27 ± 0.02 GPa, respectively (Figure 4a). The representative two-dimensional (2D) profiles of the indent impressions of both DPP-diMe and DPP-diEt single crystals along with their corresponding height profile diagrams (Figure 4b) reveal that DPP-diMe crystals are consistent with the higher share of contributions from specific C–H···O interactions perpendicular to stacked columns, as compared to the elastic DPP-diMe crystals. The lower E and H in the case of DPP-diMe are consistent with its softer nature.

To obtain further insights into the distinct mechanical response of DPP-diMe and DPP-diEt crystals, we investigated the two structures using periodic dispersion-corrected DFT methods (see supporting section 3). Upon application of the uniaxial flexural stress on the (011) face, crystals of DPP-diMe and DPP-diEt are expected to expand parallel to the a-axis on the convex (outer arc) side of the needle (Figure 5a) but compress along the concave (inner arc) side, leads to increase in angle in between molecular and (100) plane from inner arc to outer arc. These in-silico results, which can be readily accessed from the widely available computational tools, are in good agreement with the molecular level bending mechanisms proposed for elastic crystals using synchrotron microfocus X-ray diffraction studies (which are not easily accessible).

Further, we simulated the energy penalty (E_\text{p}) for applying tensile and compressive strain on both crystals in order to obtain insights into their strain tolerance behaviour along the a-axis (e_a). The resulting strain-energy and the strain-change in angle between the molecular and (100) plane curves (Figure 5b) reveal that DPP-diMe is significantly more tolerant of tensile strain than DPP-diEt. The energy barrier for tensile strain along the a-axis is only 0.23 kJ mol\(^{-1}\) for DPP-diMe upon the application of a 3% uniaxial strain along the growth axis for DPP-diMe. This is an interesting finding in our study and we do not have data to compare it with other reported molecular crystals with known mechanical properties. Nevertheless, the simulation results are in excellent agreement with the results of mechanical bending tests for DPP-diMe (Section 1 of Supporting Information, Figure S1), which showed that crystals of DPP-diMe can tolerate strains up to 6% without fracturing. By contrast, the strain-energy curve for DPP-diEt showed that an energy barrier of 3.42 kJ mol\(^{-1}\) is required to apply a 3% tensile strain on the equilibrium strain-free crystal. This energy penalty is an order of magnitude higher than that observed for DPP-diMe. Hence, our results are consistent with the brittle nature of DPP-diEt crystals. For context, this energy barrier for
applying a 3% tensile strain on crystals of DPP-diEt is of the order of the energy barrier observed in switching between crystal polymorphs. Whilst we do not have a sufficient number of published examples from DFT strain-energy calculations involving brittle and elastic molecular crystals, such a relatively large calculated energy barrier for applying a 3% strain in DPP-diEt suggests that it would not be energetically stable to withstand tensile strain during bending tests, in agreement with experiment. Both crystals display comparable energy barriers for tolerating compressive strains up to –3%. Energy frameworks calculations comparing the changes in the intermolecular interactions upon the application of crystal strain (Figure 5c and see energy frameworks discussion in section 2 & 4), reveal that DPP-diMe is stabilized by π-stacking interactions to a greater extent than DPP-diEt. This remains true irrespective of the amount of uniaxial strain applied and indeed whether this strain is tensile or compressive (Figure 5b). Upon the application of compressive strain, the π-stacking contribution to the total crystal energy becomes more stabilizing in both crystals, whilst upon the application of tensile strain, the opposite is the case (See Table S1). Nevertheless, the dominance of π-stacking interactions in DPP-diMe, combined with the low strain energy barrier along the needle axis,

![Figure 5. Suggested bending mechanism of flexible DPP-diMe crystals and the comparative strain tolerance penalties of DPP derivatives and DFT-energy profiles.](image)

(a) Bending mechanism showing expected expansion (outer arc) and compression (inner arc) along the crystal length in DPP-diMe. (b) Strain-energy curves (black symbol) and the strain-percentage of change in angle between the molecular and (100) plane (pink symbols) for DPP-diMe (blue line) and DPP-diEt (pink line), where strain is applied along the needle a-axis. (c) Energy framework contributions to the total crystal energy as a function of the intermolecular interactions observed with varying strain on the a-axis.
explains the superior mechanical flexibility of DPP-diMe.

As a proof-of-concept for the mechanical durability of these single crystals under strain and to probe the effect of the same on the semiconducting performance, we fabricated field effect transistors (FETs) by carefully sticking the individual single crystals electrostatically to lithographically patterned Au-S-D electrodes. However, the devices made using this method exhibited an extremely low channel current of ~1 pA (Figure S9) indicating poor contact between the crystal and the electrode. We could overcome the metal-semiconductor contact issues by utilizing a drop casting technique which allowed the crystals to grow on top of a flexible substrate containing lithographically patterned Au-S-D electrodes (Figure S10). PXRD measurements performed on these needle-like crystals (Figure S11) grown on top of the flexible substrates confirmed that they are identical to the single crystals used for qualitative and quantitative mechanical testing. Devices were then completed by spin coating the cytop layer and depositing Au gate electrode (Figure 6a, d; see the details in supporting information). Devices fabricated with crystals from a 2 mg/mL (1:1) dichloromethane and toluene solution of DPP-diMe exhibited a p-type current modulation with a hole field-effect mobility ($\mu_{FE}$) $\sim$ 10$^{-3}$ cm$^2$/Vs (Figure S12). Upon increasing the concentration of the DPP-diMe solution to 10 mg/mL, the density of the crystals in the channel increased beyond the percolation threshold (Figures S12, S13; details of calculation of percolation threshold are provided in supplementary section 10), resulting in an increase of channel current by two orders of magnitude and correspondingly the $\mu_{FE}$ reached a value of 0.03 cm$^2$/Vs (Figure 6c) and S14a.36,42

To ensure a proper comparison with the flexible FET of DPP-diMe, devices of DPP-diEt were also fabricated with the same concentration i.e., q 10 mg/mL but with toluene solution of DPP-diEt (ensuring > 90 % coverage of the channel area). These devices exhibited a $\mu_{FE}$ value of 0.008 cm$^2$/Vs (Figures 6g & S14b). The magnitude of mobility observed in these FETs is lower than that of DPP-diMe devices, but is comparable to many other well-known solution processable n-type and p-type semiconductors.36,38,39 This lower mobility of DPP-diEt as compared to DPP-diMe can possibly be attributed to its larger π-stacking distance (3.373 Å and 3.298 Å, respectively) between the molecular layers and smaller slipping angle (40º and 45º, respectively).34,36,42 The variation in the π-stacking distance between the molecular layers and difference in their slipping angles could be associated with the difference in the bulkiness of the alkyl side chains.40,42

![Figure 6](image)

To analyse the mechanical durability of the crystals, these flexible FETs were subjected to a bending strain parallel to the channel. Typical transfer characteristics of the flexible FETs when strained by bending the devices with different radii are shown in Figure S14a, b. Devices fabricated from both DPP-diMe and DPP-diEt based flexible FETs exhibited a decrease in the channel current as the strain radius decreased from 20 mm (strain of 0.3 %) to 5 mm (strain of 1.25 %), respectively, and consequently the $\mu_{FE}$ also decreased (Supporting Figure S14c). More importantly, the decrease in $\mu_{FE}$ is significantly higher for the DPP-diEt (~30 times decrease) compared to DPP-diMe (~5 times decrease). These observations are in excellent agreement with the inherent mechanical durability of the respective single crystals (Supporting Figure S14c), studied by the experimental and computational mechanical assessment. Despite the demonstration of mobility values as low as 10$^{-6}$ cm$^2$/Vs from the strained flexible devices fabricated from DPP-diEt, it is important to note that these measurements have been reliable over a number of devices (at least 3 devices) and such values of low mobility are not uncommon in solution processed organic semiconductors.34,36 Although it has been observed that the single crystals of DPP-diMe can withstand strains up to 3 – 6 %, we still observed a decrease in $\mu_{FE}$, for much lower strains in the device (strain range: 0.7 – 1.25 %). This behaviour can be attributed to several factors such as the difference in strain between the layers...
involved in the device fabrication, development of micro-cracks in the dielectric layer and the metal contacts with crystal. All these factors play an important role in the performance of devices in real-life applications.\textsuperscript{34-36} We further evaluated the mechanical durability of the crystals in these flexible FETs by subjecting them to multiple cycles (10 to 40 cycles with a bend radius of 5 mm) of strain parallel to the channel (Figure 6h-j). Interestingly, a device fabricated with DPP-diEt exhibited three orders of magnitude decrease in $\mu_{\text{ET}}$ after just 10 cycles of straining (Figure 6j). However, under similar conditions, the flexible FETs fabricated with DPP-diMe devices exhibited only a marginal decrease in $\mu_{\text{ET}}$ (0.019 cm$^2$/Vs to 0.014 cm$^2$/Vs) indicating the efficacy and practical utility of our molecular design in obtaining flexible electronic devices capable of reliable operation after multiple cycles of strain (Figure 6j). Notably, the performance of the DPP-diMe based flexible FETs was retained even after further bending cycles up to 40 and is projected to retain reasonable mobility even up to 275 cycles of strain, demonstrating our proof-of-concept (Figure S15). The poor durability of the DPP-diEt fabricated transistor is attributed to the brittle nature of DPP-diEt crystals. In comparison, elastic DPP-diMe crystals with higher stress tolerance can accommodate multiple cycles of strain. This behaviour is outstanding considering that our devices contain self-assembled single crystals and not the typical polycrystalline thin films (as evident from the SEM images and XRD measurements), hence demonstrating the promise of our study. Moreover, the extent of strain applied on the devices presented in this work, is significantly higher than the typical strain that a flexible wearable electronic device is expected to withstand during operation.\textsuperscript{46}

## Conclusion

In this work, we demonstrate a fully flexible field effect transistor with the two derivatives of diketopyrrolopyrrole (DPP). To the best of our knowledge, this is the first demonstration of an all-flexible FET using a flexible crystal. Moreover, we bring out a clear difference in the device behaviour when we fabricate devices from two different derivatives of the same conjugated core, one of which is flexible and the other is brittle. It was possible to demonstrate clean field effect performance for flexible FETs fabricated with both the derivatives of DPP. More importantly, flexible FETs fabricated from flexible crystal retained the field effect mobility for up to 40 bending cycles without any drop in mobility and measurable mobility value is obtained till 275 cycles of strain with a bend over a radius of 5 mm. Our calculations show a soft energy barrier for elongation along growth axis in case of DPP-diMe, with most stable structure corresponding to a non-zero positive strain. This is an interesting finding in our study and more examples of flexible crystals with calculation data will help in understanding the strain-energy correlation further. Such mechanical compliance to strain is of course missing for DPP-diEt, probably due to its brittle nature and hence we do not expect the same behaviour in the strain-energy curves for the two crystals. Our calculations also revealed the rotation of molecules in accommodating the contraction of growth axis from outer to inner arc in the bent region. This work, establishing connections from molecule to crystal to device performance, and paves the way for the fabrication of next-generation smart material that feature mechanically compliant yet crystalline active materials.

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## Keywords

Crystal engineering • Elastic semiconducting crystal • Flexible field effect transistor • Mechanical properties • Nanoindentation

### References

Elastic organic crystals of methylated diketopyrrolopyrole (DPP-diMe) and brittle crystals of ethylated diketopyrrolopyrrole (DPP-diEt) have been developed for evaluating the importance of elasticity in semiconducting organic crystals for mechanically flexible electronic devices. DPP-diMe crystals show much higher stress tolerance and retention of field-effect mobility ($\mu_{\text{FET}}$) than DPP-diEt crystals in the flexible field effect transistors, when subjected to the same flexural stress.

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