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Dihydroanthracene bridged bis-naphthopyrans: A multimodal chromophore with mechano- and photo-chromic properties

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ABSTRACT

Mechanochromophores based on bichromic molecular switches, such as bis-naphthopyranes, allow multimodal mechanochromic behavior beyond the typical binary response from single chromophores, which is important for distinguishing between multiple stress states through discrete changes in color. Spontaneously generated persistent and distinguishable multi-colors from activated bis-naphthopyranes remain challenging. And the versatility of bis-mechanophore design for advanced optical molecular systems and the fundamental insights into the corresponding mechano-reactivity are not enough. Here, we identify a dihydroanthracene bridged bis-naphthopyran as a multimodal mechanochromophore in polymers. Bridging two pyrans with the sterically constrained dihydroanthracene is helpful to control the steric effect for the favorable formation of a distinctly appreciable bis-merocyanine (bis-MC) product. By varying the length of the polymer chains, the force delivered to the mechanophore is modulated, resulting in a gradient change in the relative distribution of two distinctly colored MC products and a multicolor mechanochromism. Mechanical activation of this bis-naphthopyran proceeds via a mechanistically distinct pathway compared to the photochemical process. In addition, the bulk films can also achieve pronounced color changes when subjected to mechanical force. This study thus further expands the molecular diversity of mechanochromophores and tune the multimodal switch properties of bis-naphthopyran based polymers.

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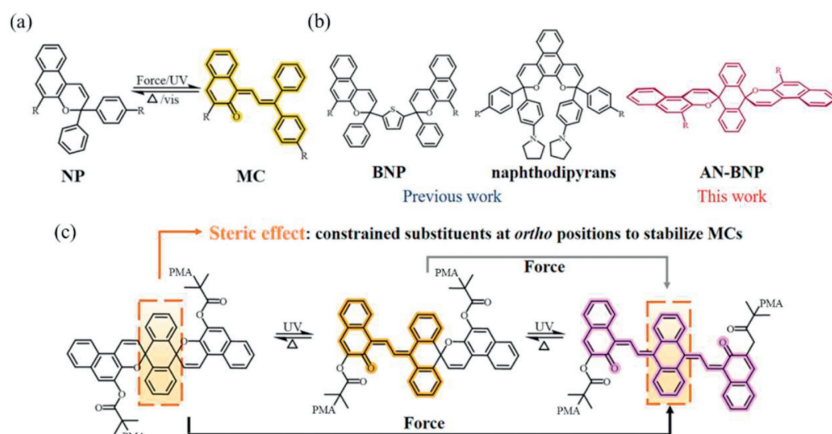
Recent decades have witnessed a rapid progress on modern polymer mechanochemistry, whereby mechanical forces are applied to promote selective chemical transformations [1–5]. Such a productive role of mechanical force is realized by introducing force sensitive groups called mechanophores into polymer backbones for efficient mechanotransduction and specific mechanochemical reactions [6–13]. So far, mechanochromic mechanophores that exhibit visually discernible coloration and/or emission in response to applied forces have been extensively explored, owing to their promising applications in stress sensing and damage detection [14–21]. Many of these mechanophores are also photochromic, e.g., spiropyran [22–28], rhodamine [29–34], oxazine [35,36]. Therefore, the corresponding dual functional polymers are versatile in switchable applications such as in storage, ophthalmic lenses, smart windows and force sensors [15,30]. Realizing on-demand chromism from

multiple responsive polymers by tuning of external force and/or light stimuli is important for advanced switches [37–40]. However, the majority of investigations relied on single chromophores displaying only simple chromism marked by binary on/off behavior. To address this limitation, multimodal mechanophores with the abilities of reversibly multiple responses and different activated states reporting are highly desirable, yet very limited [15,18,41].

Naphthopyrans (NPs) are typical chromogenic switches. Their C–O bonds are photoactivable. When embedded in a linear polymer chain, they can be converted to mechanophores which can be activated into highly colored, ring-opening merocyanine isomers (MC) under force [42–47]. Ring closure color-fading reactions are attained under visible light or thermal reversion (Scheme 1a). The photochromic and mechanochromic behaviors of the NP probes enable the straightforward visualization of encoded information, stress and/or strain in materials. Due to the easy synthetic accessibility and tunable spectrokinetic properties of NPs, diverse NP derivatives and their polymers have been developed nowadays. In particular, Robb and co-workers have successfully

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Scheme 1. (a) Transformation of naphthopyran (NP) into a colored merocyanine (MC) species is accomplished using mechanical force. (b) Examples of bis-mechanophores containing two pyrans: BNP and naphthodipyran (previous work), and AN-BNP (this work). (c) Electrochromic and reverse reactions of An-BNP produce distinctly colored MC products via photochemical, thermal, and mechanochemical process. Such unique photochromic and mechanochromic reactivities of polymeric An-BNP are powerful to distinguish between different responsive states, as a reflective of different intensity of light and/or magnitude of force.

demonstrated that synergistic coupling of two NP units into one bis-mechanophore (BNP, naphthodipyran in Scheme 1b) is a powerful strategy to achieve multicolor mechano- and photochromic properties not observed with the corresponding single NP moiety [15,38]. However, so far, the versatility of bis-mechanophore design for advanced optical molecular systems and the fundamental insights into the corresponding mechano-reactivity are not enough.

Apart from the color modulation of bis-mechanophores, controlling the thermal stability of the MCs is another crucial factor for practical applications of the bis-NP probes. Spontaneously generated persistent and distinguishable multi-colors from mono-MC intermediate and bis-MC product remain challenging, which is known as a necessity to modulate the steric effect of the NP moiety [44–49]. Intrigued by the report of stepwise photochromism of An-BNP [44], a bis-naphthopyran bridged by a dihydroanthracene, we have found An-BNP satisfied the steric requirements to achieve appreciable mechanochromism from its polymers. As demonstrated in Scheme 1, rather than electronic coupling of two pyran rings by a conjugated linker or a fused ring, herein, the two pyran units are perpendicularly oriented in the closed–closed form and the open–closed form. The di-hydroanthracene bridge furnishes constrained substituents at ortho positions to stabilize MCs in the dark [48,49], on the other hand, leads to an increase of conjugation by forming the conjugated bis-MC. Here, we explore the mechanochemical reactivity of the An-BNP mechanophore that exhibits force-dependent multicolor mechanochromism and turn-on fluorescence from its polymers both in sonicated solution and in compressed bulk state. In contrast to the preferential formation of open–closed form followed with a sequential ring-opening process that occurs with intensified light irradiation, An-BNP_{O–O} could be generated directly from An-BNP_{C–C} by force, and also via a sequential mechano-activation pathway (Scheme 1c). A dynamic equilibrium is achieved in which the distribution of MC products is increasingly biased toward An-BNP_{O–O} with greater force applied to the An-BNP mechanophore. Due to large variations in the molecular structures from An-BNP_{C–C} to An-BNP_{O–C} and An-BNP_{O–O}, drastic multiple color changes (bathochromic shift of the absorption maximum up to 100 nm between the two activated states) have been accomplished, and flexibly switched corresponding to complex on-demand (de)activations.

As shown in Scheme 2a, bis- α -bromoester functionalized An-BNP-Br was synthesized according to literature with modified procedures [44]. One of the diastereomers was separated and subjected to the following investigation, in which the pyran units

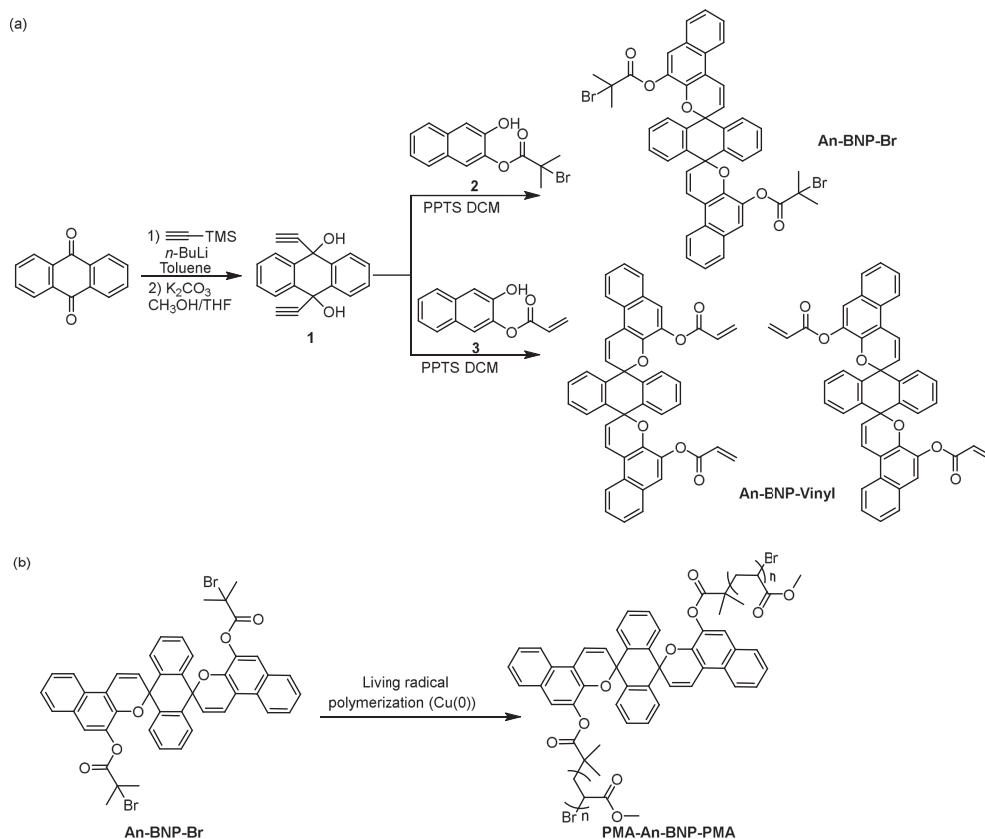
Table 1
Polymer characterization.

Polymer	M_n (kDa) ^a	D^a
PMA-An-BNP-PMA ₁₄₀	140	1.25
PMA-An-BNP-PMA ₁₂₁	121	1.11
PMA-An-BNP-PMA ₈₉	89	1.13
PMA-An-BNP-PMA ₆₈	68	1.11

^a Determined from GPC.

are connected through the central dihydroanthracene moiety in the anti-configuration. Single-electron-transfer living radical polymerization using this isomeric An-BNP-Br as the initiator and methacrylate (MA) as the monomer was conducted, offering a series of An-BNP-centered PMAs with number-average molecular weights (M_n) in the range of 68–140 kDa (Scheme 2b, Table 1). These linear polymers are well-suited for mechanochromic activation in solution, since the stress can be maximized near the chain midpoint where the labile mechanophore is located. On the other hand, for mechanochemical investigation of bulk materials, bis-acrylate functionalized An-BNP-Vinyl was synthesized in a similar protocol, which could serve as the crosslinker. Visible light induced free radical polymerization of MA in the presence of An-BNP-Vinyl (0.5 wt%) afforded a rubber-like network cross-An-BNP-PMA to bear macroscopic mechanical loads (Scheme S1 in Supporting information).

Density functional theory (DFT) calculations using the constrained geometries simulate external force (CoGEF) method were initially performed to predict the mechanochemical activity of An-BNP [49]. As shown in Fig. 1, the calculation predicts successful ring-opening reactions of both pyran units upon molecular elongation, with predicted rupture forces as 4.5 and 4.8 nN, respectively. We then compared the photochromic and mechanochromic properties of the An-BNP-centered polymers in solution. Upon UV light ($\lambda = 365$ nm, 5 W) irradiation of the polymer solution at -5 °C (PMA-An-BNP-PMA₁₄₀, 3 mg/mL in acetonitrile), the colorless solution turned yellow quickly, with absorption characteristic of the open–closed form An-BNP_{O–C} ($\lambda_{\max} = 405$ nm). A longer wavelength absorption signal appeared at ca. 505 nm under extended UV irradiation, corresponding to the formation of the highly conjugated An-BNP_{O–O} (Fig. 2a). Under constant photoirradiation, the absorbance at 405 nm increased rapidly and reached a maximum well before the absorbance at 505 nm. Such an intensity-dependent color change was in line with literature [44], and verified a sequen-



Scheme 2. (a) Synthesis of polymerizable An-BNPs (An-BNP-Br and An-BNP-Vinyl); (b) An-BNP-Br was used to initiate a living radical polymerization to give PMA-An-BNP-PMA.

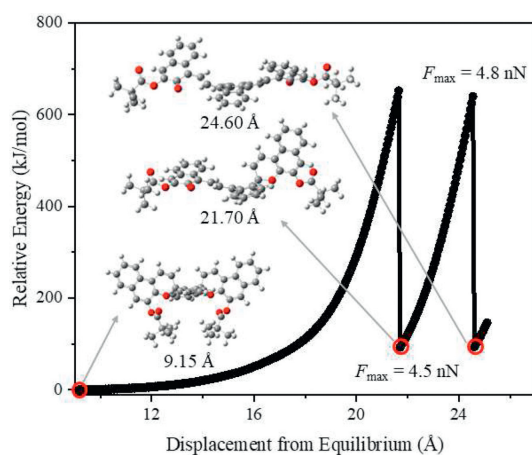


Fig. 1. CoGEF calculation (B3LYP/6-31G*) performed on truncated models of ester functionalized An-BNP, predicting the dual ring opening of both NP units upon mechanical elongation. Computed structures are shown that correspond to the force-free geometry and the product immediately after the ring-opening reactions.

tial photoactivation mechanism of the An-BNP-centered polymer with the open-closed form generated more readily.

Following, the mechanochemical reactivity of the obtained polymers was examined by ultrasonication of the polymer solutions at 0°C (3 mg/mL in acetonitrile, 1.0 s on/1.0 s off). Fig. 2b displayed the absorption spectra of sonicated PMA-An-BNP-PMA₁₄₀ acquired over time. The absorption characteristic of both An-BNP_{0-C} and An-BNP_{0-O} species were detected. Different to the sequential activation behavior under light, the two species can

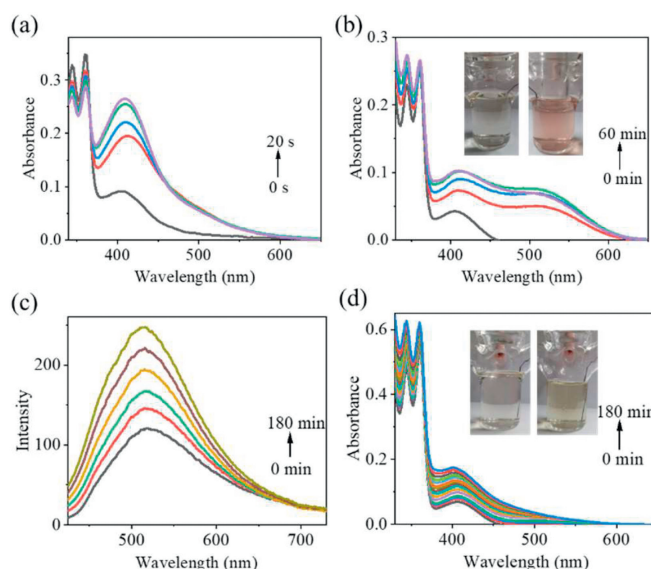


Fig. 2. Photochromic and mechanochromic properties of An-BNP-centered PMA. Absorption spectra of PMA-An-BNP-PMA₁₄₀ (a) upon exposure to UV light ($\lambda = 365 \text{ nm}$) over time, illustrating a sequential ring-opening behavior; (b) upon sonication, demonstrating a non-sequential ring-opening process and force-dependent visible absorption; (c) Fluorescence spectra ($\lambda_{\text{ex}} = 400 \text{ nm}$) of PMA-An-BNP-PMA₁₄₀; (d) Absorption spectra of PMA-An-BNP-PMA₆₈ upon sonication. Insets are photographs of polymer solutions subjected to sonication-induced mechanochemical activation: (left) before sonication, and (right) after sonication for 60 min. Polymer concentrations under sonication and under spectra record are 3 mg/mL in acetonitrile.

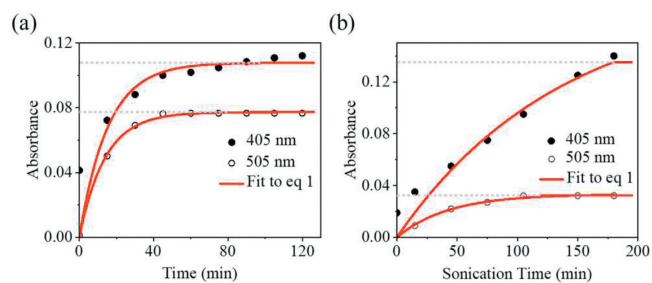


Fig. 3. Evaluation of force-dependent mechanochromism of the An-BNP mechanophore. Representative kinetic plots for (a) PMA-An-BNP-PMA₁₄₀ and (b) PMA-An-BNP-PMA₆₈, demonstrating the determination of steady-state absorbance values. Absorbance at 405 and 505 nm were recorded for each polymer during ultrasound-induced mechanical activation and fitted to Eq. 1.

emerge non-sequentially, as evidenced by the appearance of the absorbance band at 505 nm at the early stage of sonication. In addition, the absorbance at 505 nm reaches a maximum value earlier than that of 405 nm (Fig. 3). These data indicate that the bis-MC product can be formed directly from An-BNP in a relatively quicker way under mechanical forces compared to An-BNP_{O-C} [38]. It should be mentioned that non-selective polymer chain scission was also detected with extended sonication time as expected (Fig. S1 in Supporting information), leading to irreversible loss of An-BNP_{O-O}. However, it appeared to have a minimal effect on the mechanochromism [38]. Notably, alongside the orange-red coloration, sonication also led to a turn-on fluorescence of the solution, with characteristic emission peak at around 530 nm increased accordingly (Fig. 2c), possibly due to the formation of extended π -conjugation in the open-open species.

Molecular weight dependent mechanochromic reactivity was further screened with other lower molecular weight polymers (Fig. 2d, Figs. S4 and S5 in Supporting information). Taking PMA-An-BNP-PMA₆₈ for example, its solution exhibited a similar trend in absorbance over time, yet with the proportion of An-BNP_{O-C} being much larger than that of An-BNP_{O-O} (Fig. 2d). The resulted solution became less intense and exhibited yellow in color. These results suggested that the relative distribution of An-BNP_{O-C} and An-BNP_{O-O} and the correlated visible absorption spectra were force-dependent. Polymers with shorter chains are less efficient in mechanotransduction, so that the activation of An-BNP_{O-O} is overwhelming. Control experiment performed on the small molecule An-BNP-Br showed almost no changes, which additionally confirmed the mechanical origin of the observed sonication-induced reactivity (Fig. S6 in Supporting information).

To better characterize the populations of each species over time, the absorbance at 405 and 505 nm for different polymers was monitored continuously. As presented in Fig. 3 and Fig. S7 (Supporting information), for all polymers, the absorbance at 505 nm is lower than the absorbance at 405 nm. In addition, it is important to note that for polymer with higher the molecular weight, the faster it reaches its maximum absorbance at 505 nm. This finding demonstrated that the chemical equilibrium could be tuned by the applied force. This deduction was further visualized by plotting the absorbance at 405 and 505 nm as a function of sonication time until the mechanostationary state [38], in which the steady-state concentrations of An-BNP_{O-C} and An-BNP_{O-O} reached. All data of different PMAs fitted well to Eq. 1:

$$\text{Abs}(t) = B * (1 - e^{-kt}) \quad (1)$$

where Abs(t) is the absorbance at a specific wavelength at time t, B is the amplitude (the maximum absorbance value), and k is the effective rate constant. As summarized in Fig. 3 and Fig. S7, the mechanostationary state was reached faster and the relative spec-

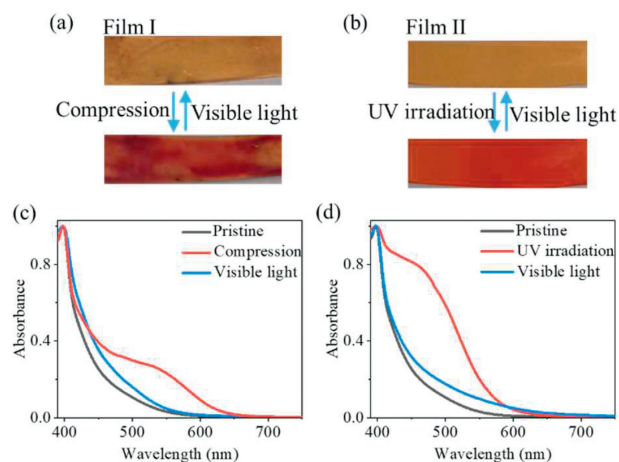


Fig. 4. (a, b) Photographs and (c, d) UV-vis absorption spectra of a mechanochemically (a, c) and photochemically (b, d) activated cross-An-BNP-PMA film. Photographs were acquired immediately after activation of force-dependent mechanochromism of the An-BNP mechanophore.

tral contribution from the An-BNP_{O-O} state becomes greater with the increase of molecular weight. In other words, a higher force can bias the chemical equilibrium to obtain more An-BNP_{O-O}, similar to those of BNP system [38].

It would be mentioned that, different to the reported observation of the fast thermal back reaction of photo-activated An-BNP, colors of the mechano-activated An-BNP-linked PMA (corresponding to both the open-open and open-close forms) are persistent, which might be attributed to the relative thermal stability of the polymeric ring-opening isomers in the dark (Figs. S8 and S9 in Supporting information) [44]. Very likely, the steric interaction derived from the dihydroanthracene bridge hinder the formation of the requisite geometric structure for ring-closure [48]. Besides, the different ratio of the MC isomers (long-lived *trans*-isomer vs. transient *cis*-isomer) formed by mechanochemical activation of polymer, compared to those generated by UV light irradiation in small molecule, might be another reason for the persistent color [48]. The main reason for the promoted thermal stability is currently under investigation. In addition, since the photochromism from the polymer is light intensity-dependent and stepwisely responsive, while the mechanical activation of An-BNP is force-dependent and proceeds *via* a mechanistically distinct pathway, tunable multimodal chromic properties from this kind of polymers were possible by fully making use of these characteristics. As illustrated in the above photo- and/or mechano-chemical experiments and summarized in Scheme 1c, different stimuli-chromic states have been observed with discrete visualized signals (including one-photon or low force activated An-BNP_{O-C}, two-photon or greater force activated An-BNP_{O-O} species and their reverting color-fading species).

Lastly, we were interested to determine if the colored MC dyes in this bis-NPs system could be generated in bulk state. When the film was subjected to mild compression of ca. 50 MPa for 1 min, a distinct orange-red color was observed (Film I, Fig. 4a). The mechanically induced color was appreciable, which could be well reserved for more than 1 months in darkness. UV-vis absorption spectrum of the activated film showed the appearance of two new absorption bands at ca. 480 and 520 nm, assignable to the open-close and open-open forms of bis-NPs, respectively, and displaying bathochromic shifts to those in solution states (Fig. 4c). On the other hand, irradiation of the elastomeric cross-An-BNP-PMA with UV light ($\lambda = 365$ nm, 10 s) produced an orange film (Film II, Fig. 4b), which was reasonably stable and could be faded rapidly with visible light exposure. The new absorption peak of the photo-activated Film II at 480 nm was more significant (Fig. 4d), which

was in contrast to that of Film I dominated with the longer wavelength band. The different coloration of the two activated films was in consistent with the experimental results in solution, and indicated that the concentration of MC and bis-MC species in bulk state could be tuned by different kinds and magnitude of stimuli.

In conclusion, we have developed a dihydroanthracene bridged bis-naphthopyrans (An-BNP) mechanophore that exhibited force-dependent, multicolor mechanochromism in polymers. The sterically constrained bridge was helpful to control steric effect of the mechanochromic ring-opening reactions, leading to persistent colors from activated polymers both in sonicated solutions and compressed bulk films. Increasing the force applied to the An-BNP mechanophore by increasing the length of the attached polymer chains could gradiently alter the relative proportion of the two MC products, as well as vary the color of the solution between yellow and orange-red. Moreover, mechanical activation of An-BNP proceeded *via* a mechanistically distinct pathway compared to the photochemical process. Orthogonally usage of the disparate intensity-dependent photochromic and force-dependent mechanochromic reactivities allowed multimodal chromism from the obtained polymers. Our work thus enriches the versatility of the bis-mechanophore design, which will open up great possibilities to tune multimodal switch properties of mechanochromic polymers. Applications of this multimodal mechanochromophore to distinguish between multiple stress states with increased sensitivity are undergoing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2023.108949.

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