

Electronic Supplementary Information

**Reconfigurable Crosslinking System via Asymmetric Metal-Ligand Coordination Strategy**

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## Materials and Methods

All reagents were commercially available and used as supplied without further purification. *p*-Phenylenediamine (97%, SCR Ltd.), Maleic anhydride (98%, Nanjing CR Ltd.), Sulfur (98%, Nanjing CR Ltd.), Malonyl dichloride (96%, Aladdin chemical), Polypropylene glycol bis(2-aminopropyl ether) ( $M_n = 2000$ , Aladdin chemical), Lawesson reagent (98%, Meryer chemical), Dimethyl sulfoxide (DMSO, 98%, Energy chemical), Triethylamine (TEA, 99.5%, Energy chemical), Aniline (99%, Energy chemical), Copper(II) chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ , 99.9%, Aladdin chemical), Manganese(II) chloride ( $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ , 99.9%, Aladdin chemical), Europium(III) chloride ( $\text{EuCl}_3 \cdot 6\text{H}_2\text{O}$ , 99.9%, Aladdin chemical). Deuterated solvents were purchased from Cambridge Isotope Laboratory (Andover, MA).

$^1\text{H}$  NMR spectra were recorded on a Bruker DRX 400 NMR spectrometer at room temperature with use of the deuterated solvent and the residual solvent or TMS as the internal reference (DMSO- $d_6$ ,  $\delta$  2.50 ppm for  $^1\text{H}$ ).

Infrared radiation spectra were recorded with Nicolet iS10 infrared spectrometer.

TEM photography was recorded with JEM-1011 transmission electron microscopy.

GPC was performed at a flow rate of 1.0 mL/min with tetrahydrofuran (THF) as the moving phase on a PL-GPC 120 system equipped with a refractive index (RI) detector, and narrowly dispersed polystyrene species were employed as the molecular weight standard.

The *in situ* XPS analysis was performed on a PHI 5000 VersaProbe system, using monochromatic Al K $\alpha$  radiation (1486.6 eV). The binding energies were corrected by setting the graphite carbon C 1s signal of 284.6 eV as reference.

**Isothermal Titration Calorimetry (ITC).** All titrations were performed using a MicroCal ITC200 isothermal titration calorimeter at 298.15 K. The solutions were prepared in dry MeOH using vacuum-dried hosts and guests. Twenty aliquots (2  $\mu\text{L}$  each) of inorganic salt in dry MeOH were added into the ligand solution with 150 s injection interval to measure the heat of complexation. Blank titrations in dry MeOH were performed and subtracted from the corresponding titrations to remove the effect of dilution. The titration curve thus obtained was analyzed using ORIGIN software, giving parameters concerning the complex stoichiometry (N), the binding affinity (K) and thermodynamic parameters ( $\Delta H$  and  $\Delta S$ ) of different titrations.

The **small-angle x-ray scattering (SAXS)** measurements were performed on Beamline 4-2 at Stanford Synchrotron Radiation Lightsource (SSRL) of SLAC National Accelerator Laboratory (SLAC). The incident x-ray beam with an energy of 15 keV was used, and it was normal to the sample film so that the SAXS was in transmission geometry. A Rayonix MX225-HE CCD x-ray detector was used, with a sample-to-detector distance of 3489.2 mm. The 2D CCD images were then reduced into scattering intensity (I) as a function of scattering vector ( $q = 4\pi\sin\theta/\lambda$ , where  $\theta$  is half of the scattering angle and  $\lambda$  is x-ray wavelength.) using the software equipped at the beamline.

All the **single-molecule force spectroscopy** experiments were performed on a commercial AFM (NanoWizard IV from JPK Instruments AG, Germany). The force-extension curves are recorded by JPK Data processing software and further analyzed by customer written procedure based on Igor pro 6.12 (Wavemetrics. Ins, USA). Soft silicon nitride cantilevers (MLCT, D type, Bruker, USA) were used in all AFM tests. First, 200  $\mu\text{l}$  of *compounds solution* was deposited on a piranha treated glass slide for 30 minutes to allow the compounds nonspecifically absorb on substrates surface. Then the substrates were rinsed with excessive deionized water to remove the unattached compounds. Next, the fluid chamber was filled with 2 ml of DMF and waited 30 minutes to let the system reach equilibrium state. In a typical force measurement, the cantilever was approached to the

substrates at a constant speed of  $1000 \text{ nm s}^{-1}$  and held on the substrates at 3 nN for 3 seconds to establish the nonspecific interactions between cantilever and compounds. The nonspecific interaction between compounds and substrates or cantilever surface is strong enough to hold the compounds. Then the cantilever was retracted at the same speed. In  $\sim 3\%$  of total force curves, saw-tooth like force-extension curves were observed. Each rupture force peak was fitted by worm-like chain model.

The **rheology** experiments were tested on Discovery HR-2 TA rheometer. For frequency-sweep experiment, 1% strain in the linear region of the sample was adopted at  $25 \text{ }^\circ\text{C}$ . Master curves of the dynamic modulus were shifted by time temperature superposition (TTS).

**Mechanical tensile-stress experiments** were performed using an Instron 5944 Microtester. For mechanical tensile-stress and self-healing test, sample size of 10 mm length  $\times$  5 mm width  $\times$  1 mm height, gauge length of 3mm, and strain rate of  $20 \text{ mm}\cdot\text{min}^{-1}$  were adopted. All samples were tested at ambient conditions.

The hysteresis is measured by subjecting a sample containing no crack to load and unload. And the hysteresis is defined by  $W_D/W$ , where  $W$  is the area under the stress-stretch curve measured on loading, and  $W_D$  is the area between the loading and unloading curves.

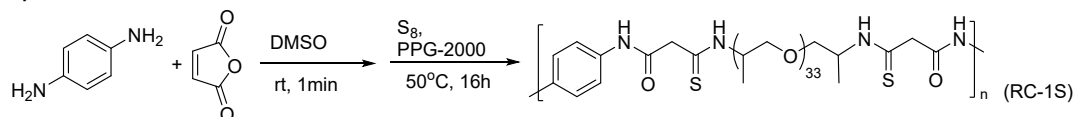
For self-healing tests, the samples were cut into two completely separate pieces with a razor blade. The two half films were gently brought back into contact with a distance about  $\sim 40 \text{ }\mu\text{m}$  and then healed at  $25^\circ\text{C}$  with one drop of DMF for different time.

The principal scheme of **polarized FT-IR spectroscopy measurements** is illustrated in Figure S19. A film sample under investigation was uniaxially drawn and recovered in a miniaturized stretching machine, which was modified to fit in the sample compartment of the spectrometer. During deformation, 16-scan interferograms were acquired in small time intervals with polarized radiation alternately changed between the parallel and perpendicular directions with respect to the machine axis. The typical sample thickness for FTIR experiments was about  $30 \text{ }\mu\text{m}$  measured by a micrometer. Great caution was taken to trim the samples to form the dimensions of approximately  $10 \times 5 \text{ mm}^2$ .

The **X-ray absorption fine structure** (XAFS) spectra were acquired at the 1W2 beamline of Beijing Synchrotron Radiation Facility (BSRF) under the fluorescence mode. The energies were calibrated accordingly to the absorption edge of pure Cu and Mn foils. Athena and Artemis codes were used to extract the data and fit the profiles. For the X-ray absorption near edge structure (XANES) part, the experimental absorption coefficients as a function of energies  $\mu(E)$  were processed by background subtraction and normalization procedures and reported as “normalized absorption” with  $E_0 = 8979.0 \text{ eV}$  for all the tested samples and Cu foil/CuO/Cu<sub>2</sub>O standard.

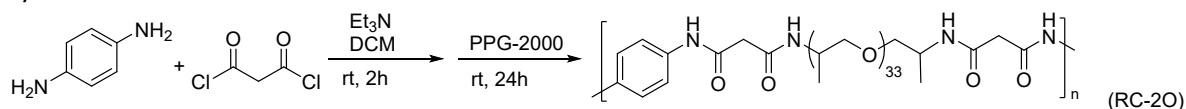
## Synthesis Procedures

### Synthesis of RC-1S



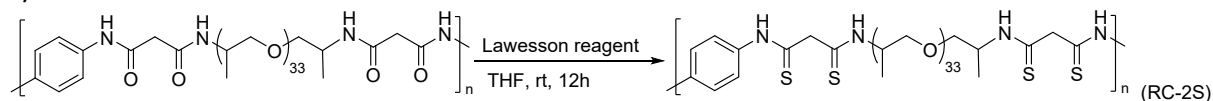
The polymer RC-1S was prepared by following a modified literature report<sup>1</sup>. *p*-Phenylenediamine (1 mmol, 108 mg) and DMSO (0.6 mL, 9 equiv) was added into a 10 mL test tube containing solid anhydride maleic (2 mmol, 196 mg). The resulting mixture was shaken vigorously with a vortex mixer (5 min) to give a viscous pale yellow. Then polypropylene glycol bis(2-aminopropyl ether) (1 mmol, 2 g) sulfur (2.5 mmol, 80 mg) and a magnetic stir bar were added. The tube was closed with a septum and stirred at 50°C for 16 h. After reaction, the solution was concentrated in vacuum and 10 mL hexane was poured into it. dark-yellow precipitate-like viscous liquid appeared and the mixture was settled for half an hour. The upper clear solution was then decanted. The dissolution-precipitation-decantation process was repeated for three times and the final product was subjected to vacuum evaporation to remove the solvent, achieving dark-yellow oil. Yield: 87%. Molecular weight according to GPC:  $M_w = 36252$ ;  $M_n = 13276$  ( $\bar{D} = 2.73$ ). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  10.10 (s, 1H), 10.06 (s, 1H), 7.82 (s, 2H), 7.52 (s, 4H), 3.70 (s, 4H), 3.60-3.38 (m, 94H), 1.03 (d, *J* = 5.8 Hz, 104H) (Fig. S1).

### Synthesis of RC-2O



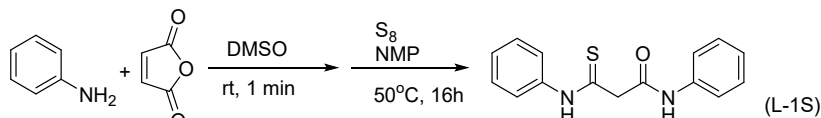
Malonyl dichloride (2 mmol, 282 mg) and triethylamine (5 mmol, 505 mg) were dissolved in 10 mL anhydrous DCM under N<sub>2</sub> atmosphere, cooling to 0°C. Then *p*-phenylenediamine (1 mmol, 108 mg) in DCM solution was dropwise added into the tube for 0.5 h. The reaction was stirred at 0°C for 2 h, after which polypropylene glycol bis(2-aminopropyl ether) (1 mmol, 2 g) in DCM solution was added. Then the reaction was stirred at room temperature for 24 h. 50 mL H<sub>2</sub>O was added to quenching reaction, using DCM to extract product. The organic layer was washed by HCl solution, NaHCO<sub>3</sub> solution and brine. then organic layer was subjected to vacuum evaporation to remove the solvent, achieving yellow oil. Yield: 76%. Molecular weight according to GPC:  $M_w = 22538$ ;  $M_n = 12806$  ( $\bar{D} = 1.76$ ). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  7.92-7.84 (m, 5H), 7.65-7.49 (m, 2H), 7.26-7.15 (m, 1H), 3.55 (s, 4H), 3.50-3.40 (m, 97H), 1.03 (d, *J* = 5.7 Hz, 102H) (Fig. S2).

### Synthesis of RC-2S



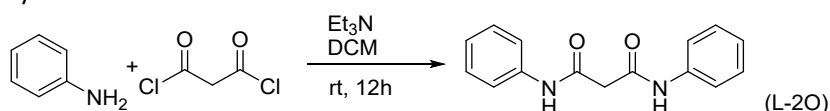
The polymer RC-2S was prepared by following a modified literature report<sup>2</sup>. RC-2O (3 g) and Lawesson reagent (1 g) were added into tube, dissolved by 50 mL THF. The reaction was stirred at room temperature for 12 h. After reaction, the solution was concentrated in vacuum and 100 mL hexane was poured into it. yellow precipitate-like viscous liquid appeared and the mixture was settled for half an hour. The upper clear solution was then decanted. The dissolution-precipitation-decantation process was repeated for three times and the final product was subjected to vacuum evaporation to remove the solvent, achieving yellow oil. Yield: 78%. Molecular weight according to GPC:  $M_w = 22784$ ;  $M_n = 13986$  ( $\bar{D} = 1.63$ ). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  7.78-7.62 (m, 4H), 7.02-6.76 (m, 4H), 3.54-3.27 (m, 101H), 1.04 (d, *J* = 5.7 Hz, 100H) (Fig. S3).

### Synthesis of L-1S



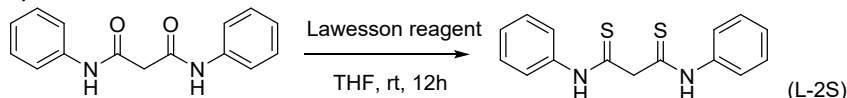
The polymer L-1S was prepared by following a literature report<sup>1</sup>. Aniline (2 mmol, 186 mg) and DMSO (0.3 mL, 4.5 equiv.) was added to a 7-mL test tube containing solid anhydride maleic. The resulting mixture was shaken vigorously with a vortex mixer to give a viscous pale yellow solution. Sulfur (40 mg, 1.25 mmol), *N*-methylpiperidine (99 mg, 1 mmol) (used for aniline derivatives) and a magnetic stir bar were added. The tube was closed with a septum, and the tube was stirred at 50°C for 16 h. The reaction mixture was purified by column chromatography on silica gel (eluent CH<sub>2</sub>Cl<sub>2</sub>:EtOAc 1:0 to 20:1) to afford product as a pale yellow solid (168 mg, 62%). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>): δ 10.51 (s, 1H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.61-7.48 (m, 1H), 7.38-7.26 (m, 3H), 7.18-7.02 (m, 3H), 6.72-6.57 (m, 2H) (Fig. S4).

#### Synthesis of L-2O



Aniline (2 mmol, 186 mg) and triethylamine (2.5 mmol, 253 mg) were dissolved in 20 mL anhydrous DCM under N<sub>2</sub> atmosphere, cooling to 0°C. Then Malonyl dichloride (1 mmol, 141 mg) in DCM solution was dropwise added into the tube for 0.5h. The reaction was stirred at room temperature for 12h. 50 mL H<sub>2</sub>O was added to quenching reaction, using DCM to extract product. The organic layer was washed by HCl solution, NaHCO<sub>3</sub> solution and brine. The reaction mixture was purified by column chromatography on silica gel (eluent CH<sub>2</sub>Cl<sub>2</sub>) to afford product as a pale yellow solid (201 mg, 79%). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>): δ 10.15 (s, 2H), 7.60 (d, *J* = 7.7 Hz, 4H), 7.32 (t, *J* = 7.7 Hz, 4H), 7.06 (t, *J* = 7.3 Hz, 4H), 3.47 (s, 2H) (Fig. S5).

#### Synthesis of L-2S



The polymer L-2S was prepared by following a modified literature report (2). L-2O (127 mg) and Lawesson reagent (200 mg) were added into tube, dissolved by 5 mL THF. The reaction was stirred at room temperature for 12h. The reaction mixture was purified by column chromatography on silica gel (eluent CH<sub>2</sub>Cl<sub>2</sub>) to afford product as a pale yellow solid (127 mg, 88%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 9.42 (s, 2H), 7.55 (d, *J* = 7.7 Hz, 4H), 7.32 (t, *J* = 7.7 Hz, 4H), 7.15 (t, *J* = 7.3 Hz, 4H), 3.82 (s, 2H) (Fig. S6).

#### Synthesis of RC-1S-Mn film

RC-1S (1 mmol, 2.28 g) and MnCl<sub>2</sub>·4H<sub>2</sub>O (0.2 mmol, 40 mg) were dissolved in 10 mL DMF. Then the reaction was stirred at 85°C for 12h and then concentrated. The concentrated solution was poured into a PTFE mold and dried at room temperature for two days followed by drying at 70 °C for 12 h. The polymer film was then peeled off from the PTFE mold for further testing.

#### Synthesis of RC-2O-Mn film

RC-2O (1 mmol, 2.22 g) and MnCl<sub>2</sub>·4H<sub>2</sub>O (0.2 mmol, 40 mg) were dissolved in 10 mL DMF. Then the reaction was stirred at 85°C for 12h and then concentrated. The concentrated solution was poured into a PTFE mold and dried at room temperature for two days followed by drying at 70 °C for 12 h. The polymer film was then peeled off from the PTFE mold for further testing.

#### Synthesis of RC-2S-Mn film

RC-2O (1 mmol, 2.34 g) and  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (0.2 mmol, 40 mg) were dissolved in 10 mL DMF. Then the reaction was stirred at  $85^\circ\text{C}$  for 12h and then concentrated. The concentrated solution was poured into a PTFE mold and dried at room temperature for two days followed by drying at  $70^\circ\text{C}$  for 12 h.

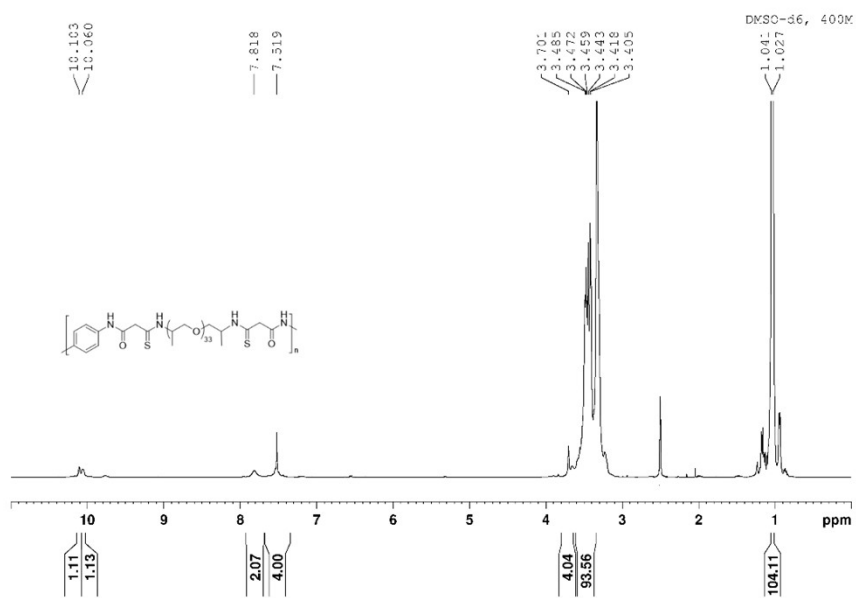
#### Synthesis of RC-1S-Cu film

RC-1S (1 mmol, 2.28 g) and  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.2 mmol, 34 mg) were dissolved in 10 mL DMF. Then the reaction was stirred at  $85^\circ\text{C}$  for 12h and then concentrated. The concentrated solution was poured into a PTFE mold and dried at room temperature for two days followed by drying at  $70^\circ\text{C}$  for 12 h. The polymer film was then peeled off from the PTFE mold for further testing.

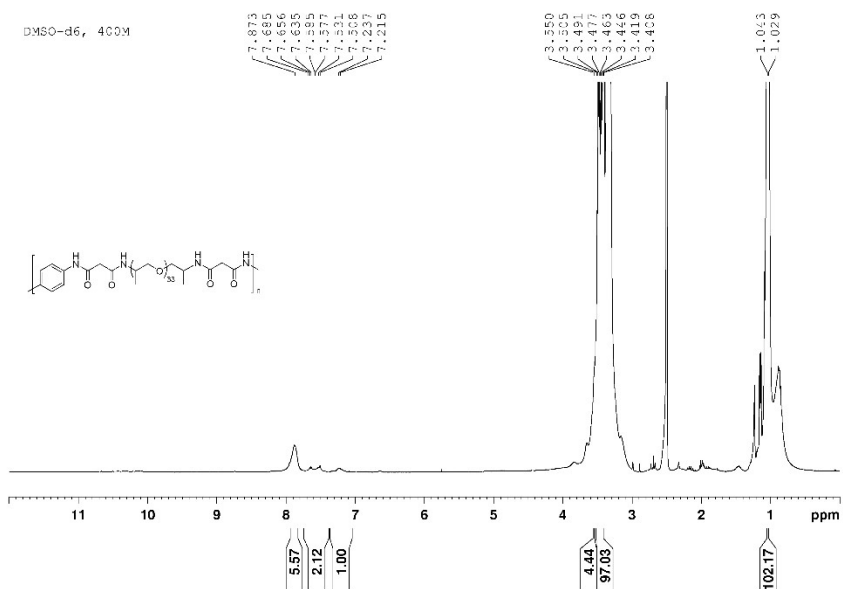
#### Synthesis of RC-1S-Eu film

RC-1S (1 mmol, 2.28 g) and  $\text{EuCl}_3 \cdot 6\text{H}_2\text{O}$  (0.2 mmol, 73mg) were dissolved in 10 mL DMF. Then the reaction was stirred at  $85^\circ\text{C}$  for 12h and then concentrated. The concentrated solution was poured into a PTFE mold and dried at room temperature for two days followed by drying at  $70^\circ\text{C}$  for 12 h. The polymer film was then peeled off from the PTFE mold for further testing.

## Results and Data



**Figure S1.**  $^1\text{H}$  NMR spectrum of compound RC-1S (DMSO- $d_6$ , room temperature, 400 MHz)



**Figure S2.**  $^1\text{H}$  NMR spectrum of compound RC-20 (DMSO- $d_6$ , room temperature, 400 MHz)

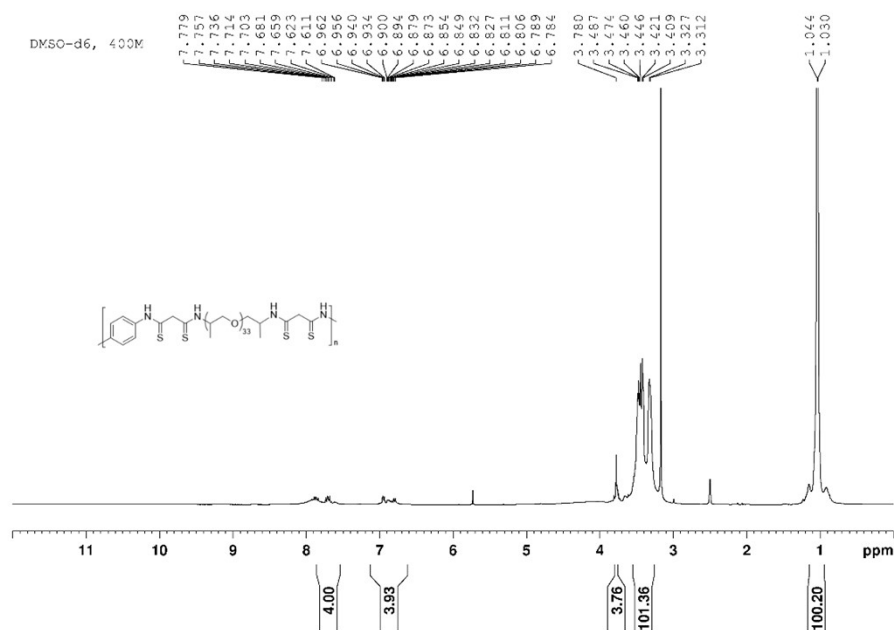


Figure S3. <sup>1</sup>H NMR spectrum of compound RC-2S (DMSO-*d*<sub>6</sub>, room temperature, 400 MHz)

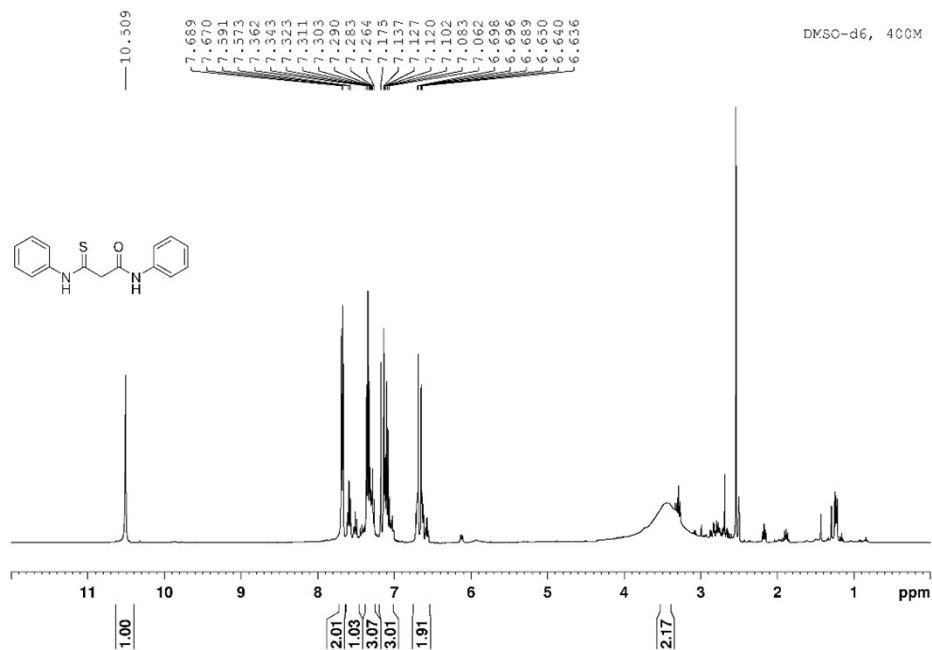


Figure S4. <sup>1</sup>H NMR spectrum of compound L-1S (DMSO-*d*<sub>6</sub>, room temperature, 400 MHz)



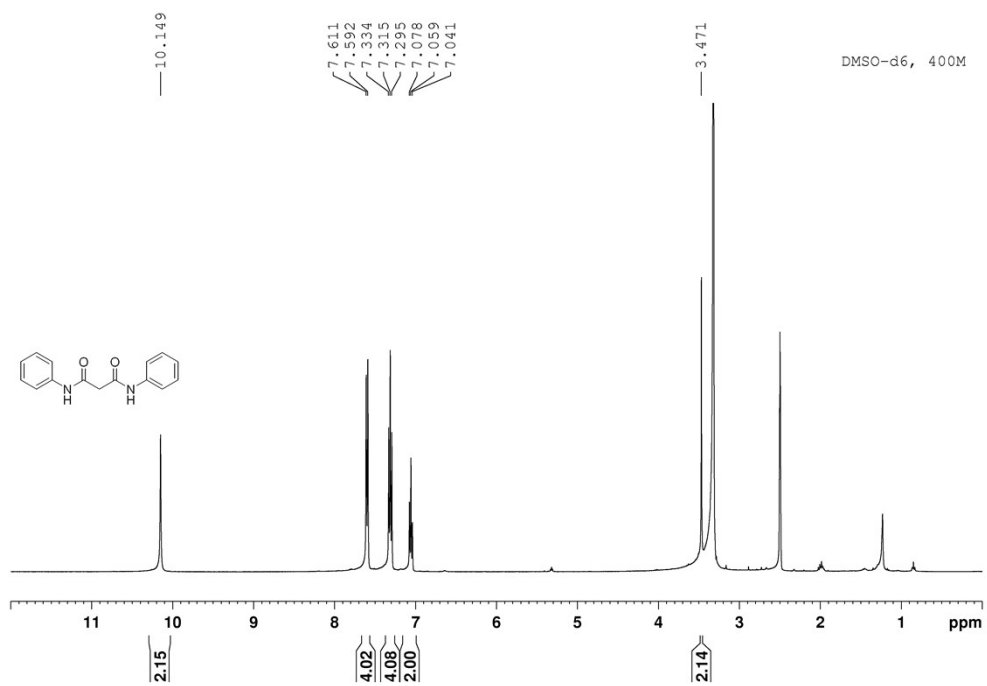


Figure S5. <sup>1</sup>H NMR spectrum of compound L-2O (DMSO-*d*<sub>6</sub>, room temperature, 400 MHz)

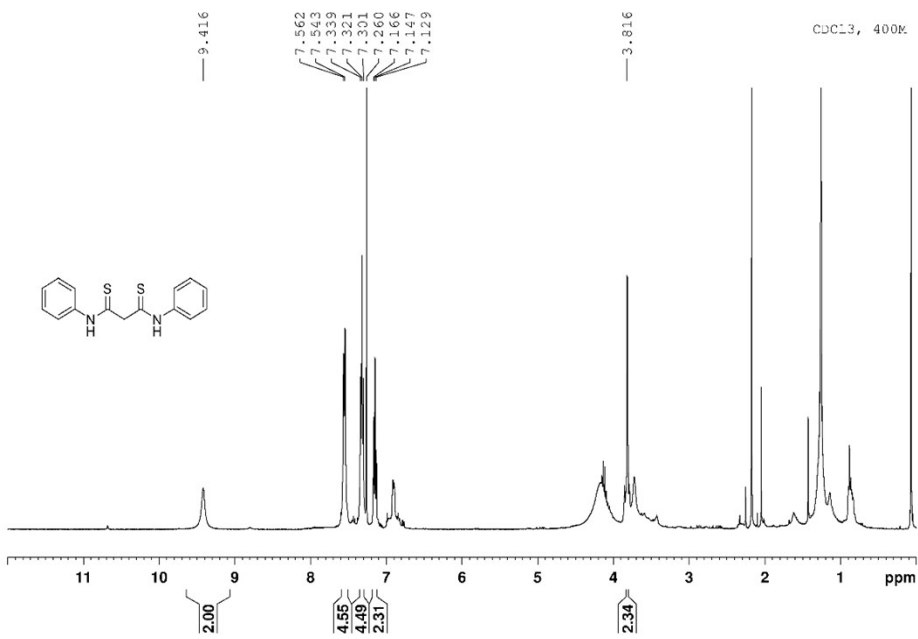
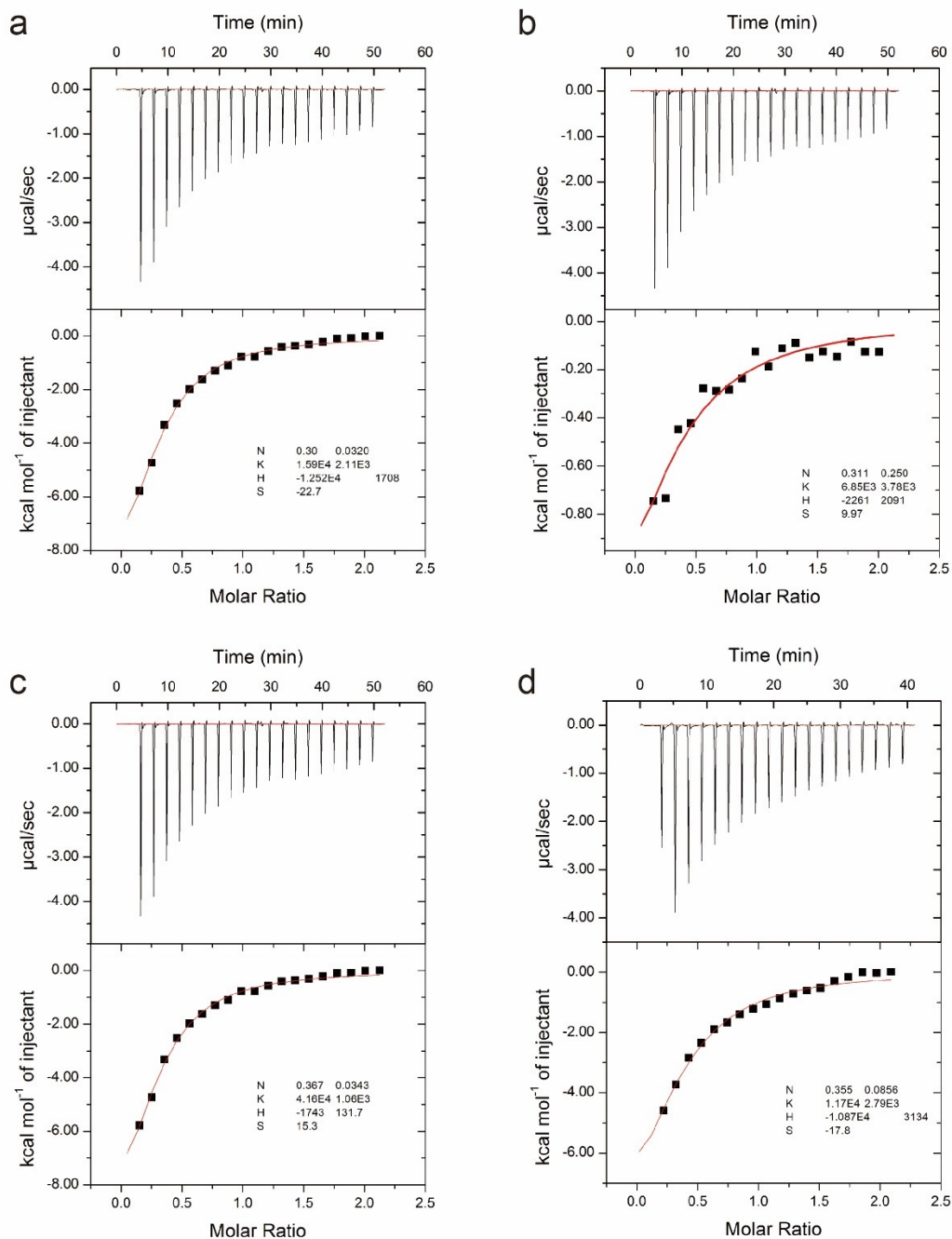
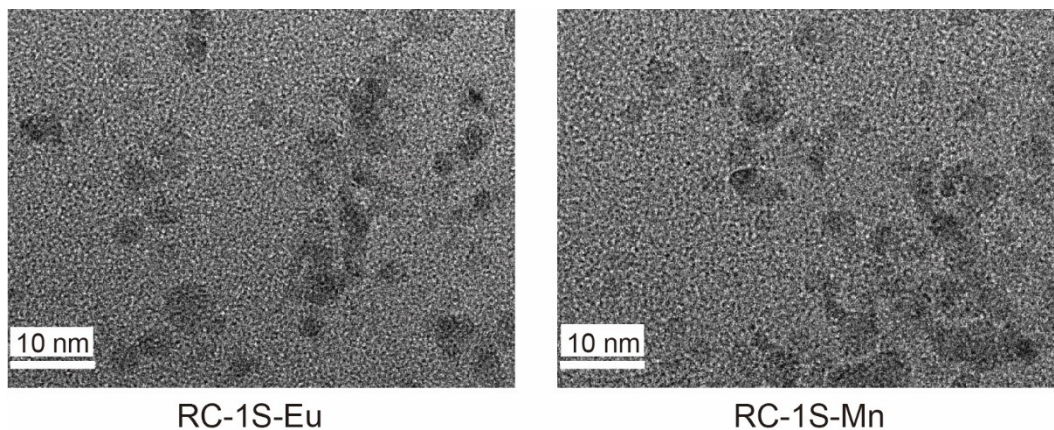


Figure S6. <sup>1</sup>H NMR spectrum of compound L-2S (DMSO-*d*<sub>6</sub>, room temperature, 400 MHz)

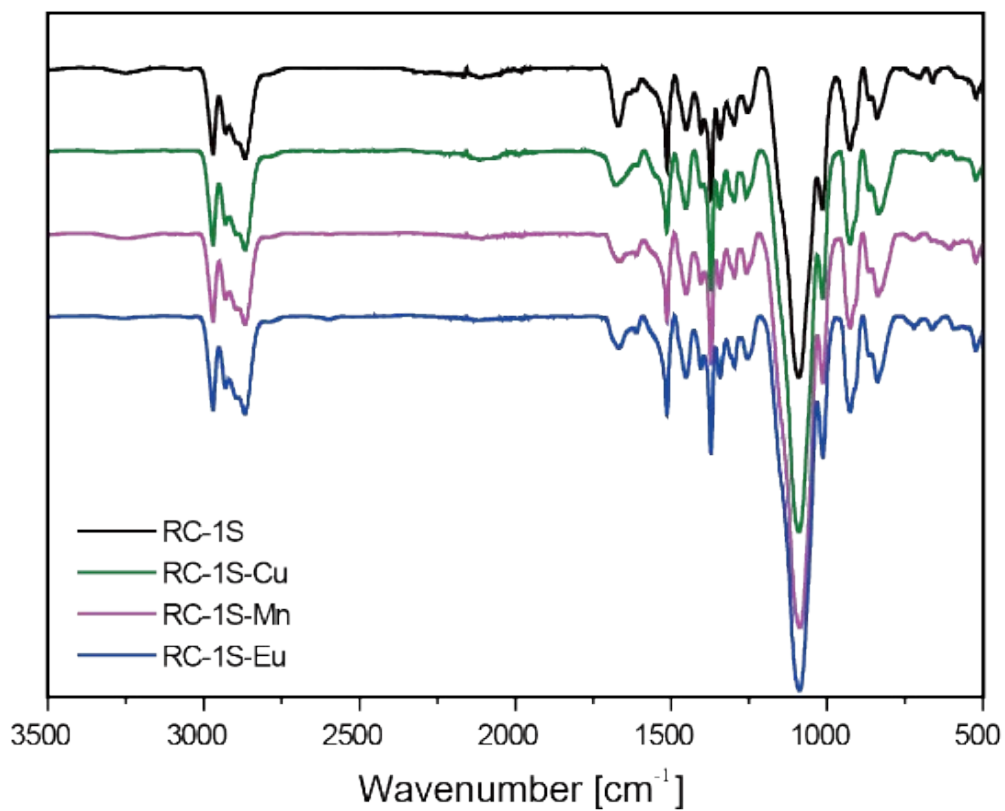
**Table S1.** characters of S, O atoms

Atom	Electronegativity	Radius
Sulfur	2.58	1.04 Å
Oxygen	3.44	0.66 Å

**Figure S7.** ITC titration data of all samples. (a) L-1S (0.3 mM, in cell) with MnCl<sub>2</sub> (3 mM, in syringe), (b) L-2O (0.3 mM, in cell) with MnCl<sub>2</sub> (3 mM, in syringe), (c) L-1S (0.3 mM, in cell) with CuCl<sub>2</sub> (3 mM, in syringe), (d) L-1S (0.3 mM, in cell) with EuCl<sub>3</sub> (3 mM, in syringe) in MeOH at 298 K.



**Figure S8.** TEM images of dynamic interaction system in the RC-1S-Eu/Mn substrate with scale bar of 10 nm.



**Figure S9.** FT-IR of samples with different cations in the 500-3500 $\text{cm}^{-1}$  ( $\text{Cu}^{2+}$ ,  $\text{Mn}^{2+}$  and  $\text{Eu}^{3+}$ ).

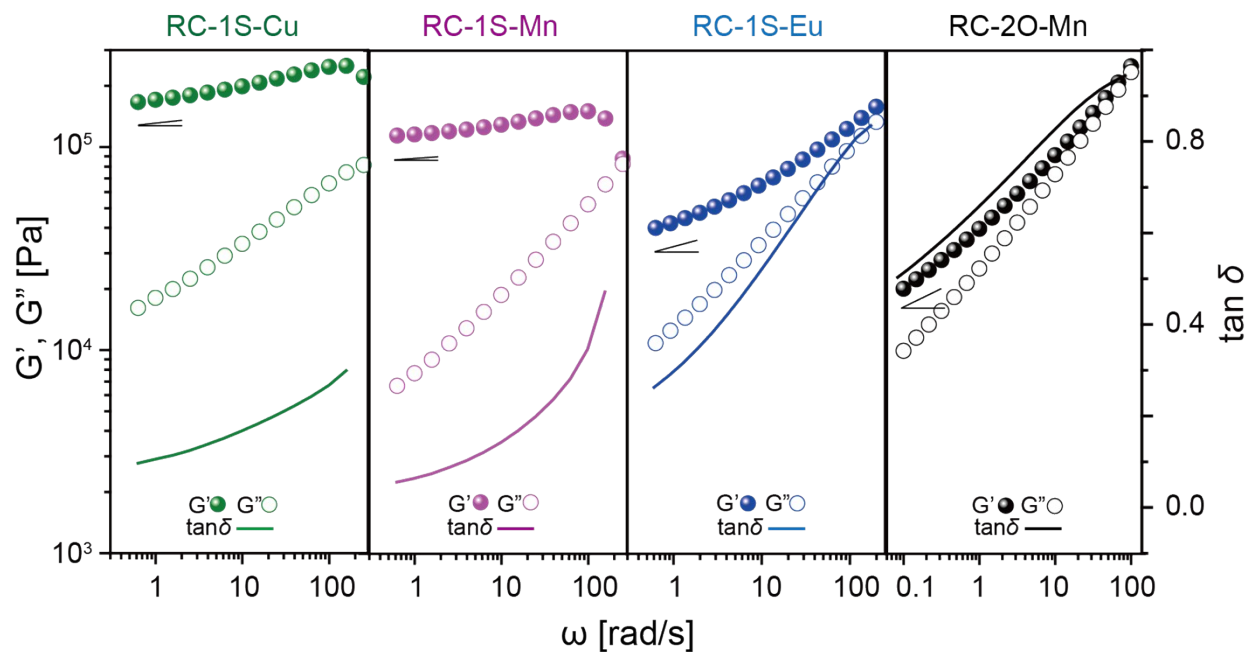


Figure S10. the plateau modulus  $G_{pl}$  determined from the value of  $G'$  at the frequency of the  $\tan \delta$  minimum.

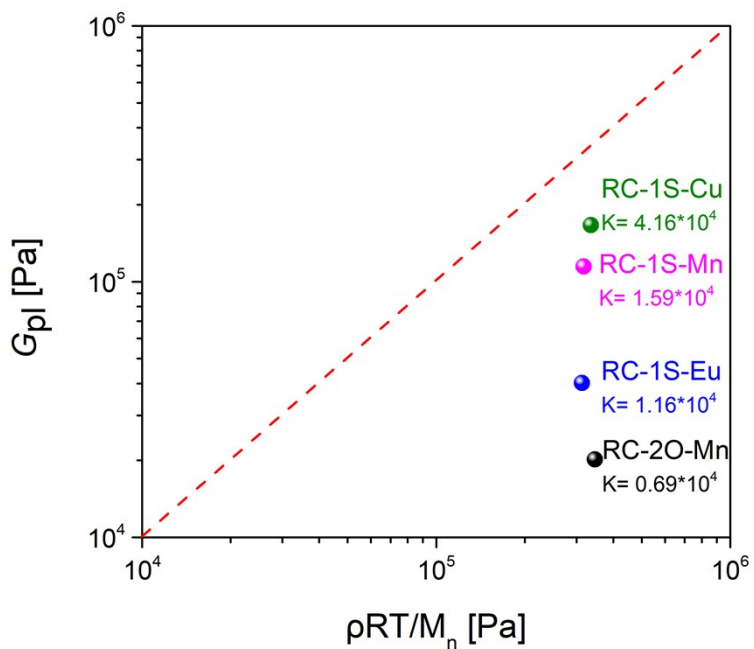
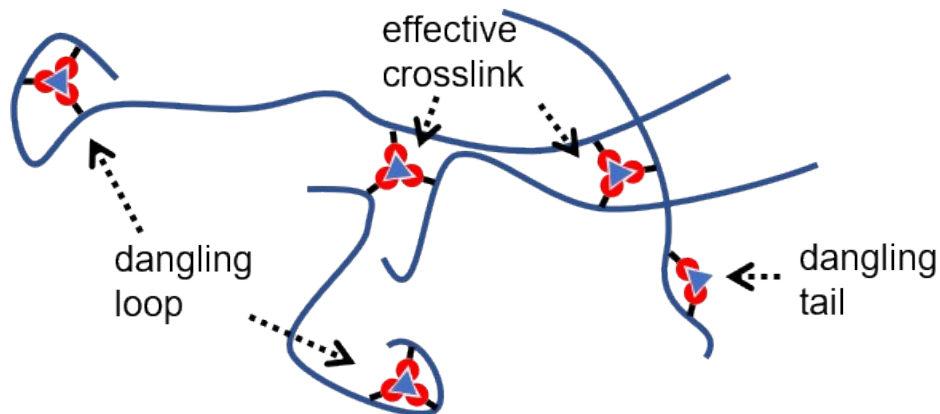
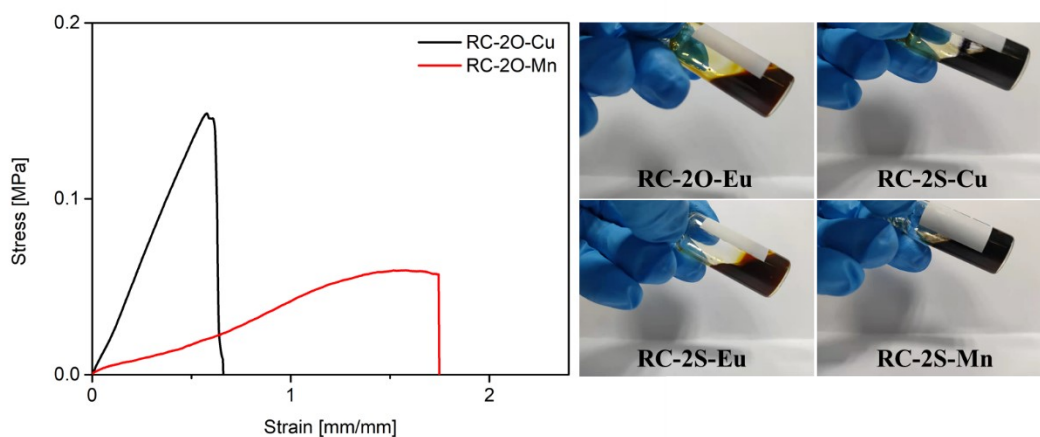


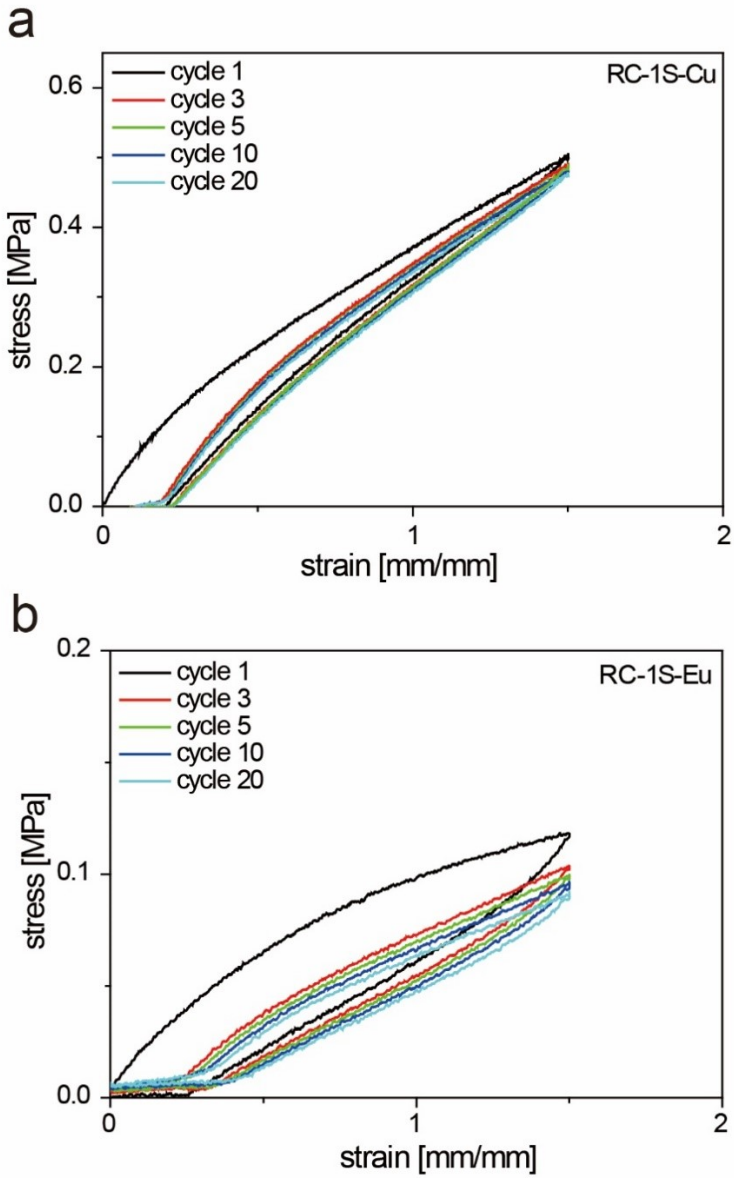
Figure S11. Plots of the plateau modulus  $G_{pl}$  against  $\rho RT/M_n$  of samples, with different binding ability  $K$ .



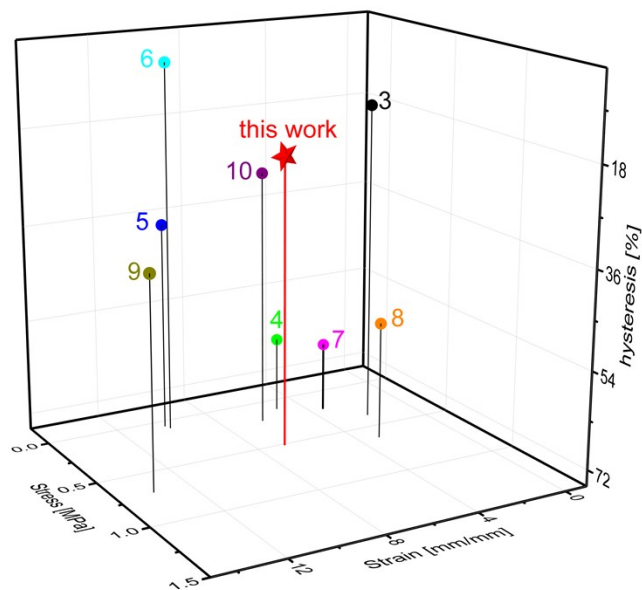
**Figure S12.** Schematic illustration of the network sustained by aggregates of coordination. The skeleton may carry effective crosslink, dangling loop and dangling tail.



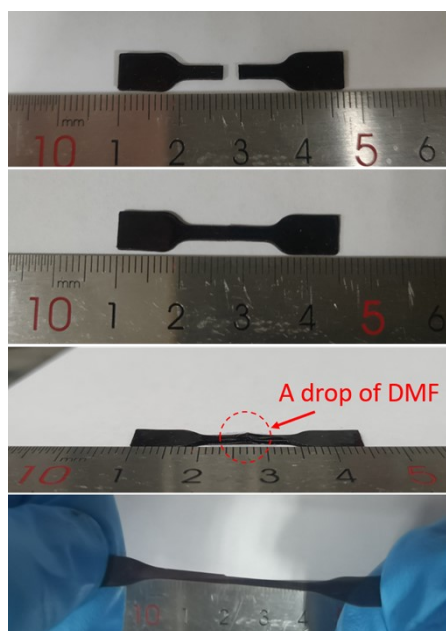
**Figure S13.** Stress-strain curves of RC-20-Cu/Mn, while RC-20-Eu and RC-2S-Cu/Mn/Eu were viscous liquids.



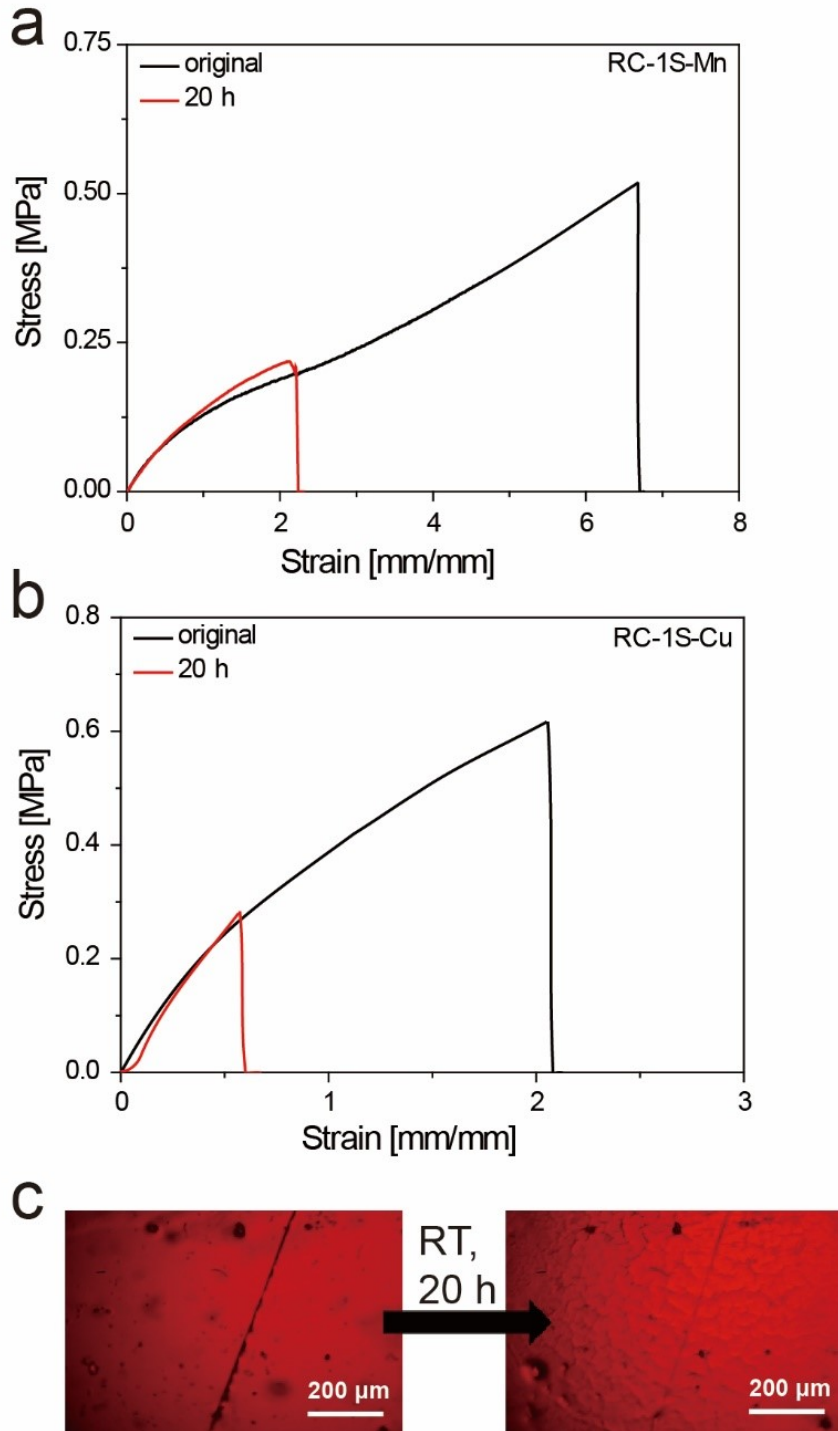
**Figure S14.** Cyclic loading and recovery of samples. (a) RC-1S-Cu, (b) RC-1S-Eu. The samples were conducted cyclic loading for 20 times (150% strain) at a tensile rate of  $10 \text{ mm} \cdot \text{min}^{-1}$ .



**Figure S15.** Comparison of tensile stress, tensile strain and hysteresis with recently reported elastomers based on supramolecular or noncovalent-bonded strategies (hysteresis ratio is estimated according to the data reported in the reference)<sup>3-10</sup>.

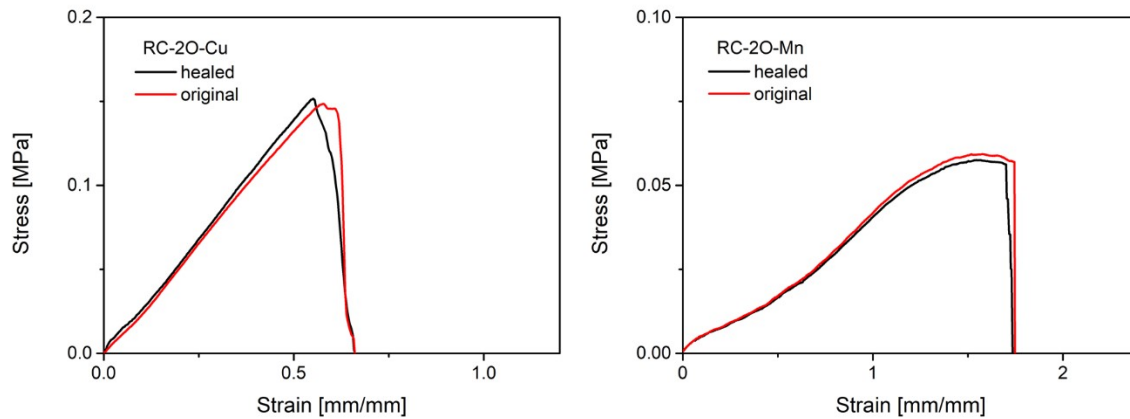


**Figure S16.** Typical self-healing process of RC-1S-Eu with the assist of DMF solvent: the cut pieces were aligned and joined together, and we added a drop of DMF for wetting onto the wound. After repaired for 20 hours at room temperature, the sample was healed and the stretchability restored.

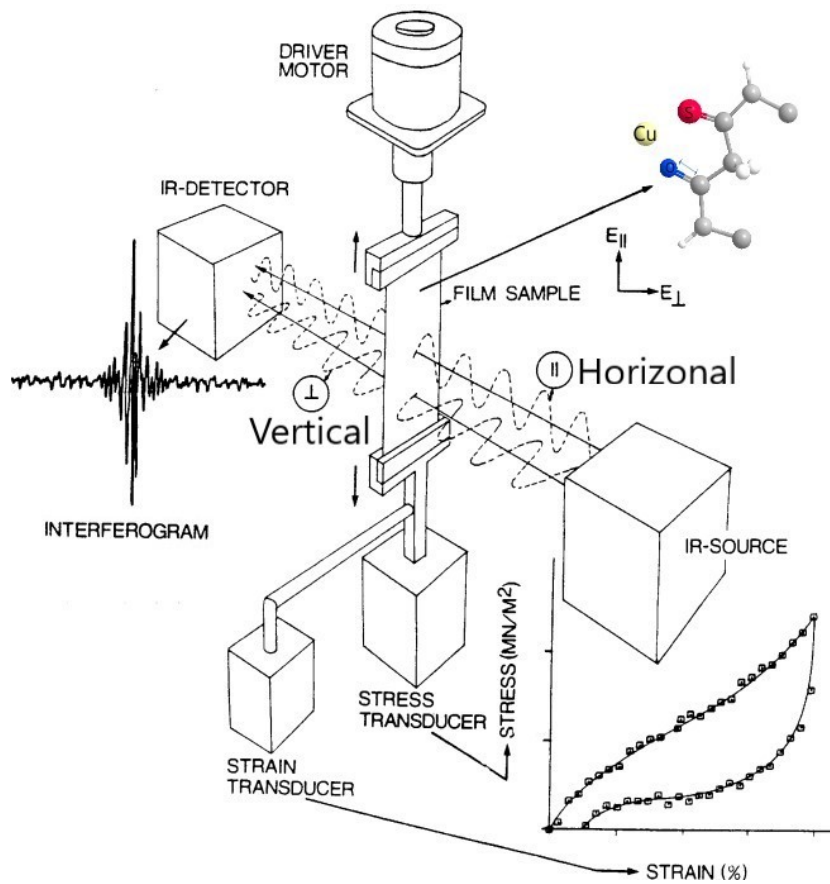


**Figure S17.** Self-healing tests of samples. (a) Stress–strain curves of the original and healed RC-1S-Mn specimens after healing for 20 h at room temperature. (b) Stress–strain curves of the original and healed RC-1S-Cu specimens after healing for 20 h at room temperature. (c) The sample was cut into two pieces which were joined together for healing at room temperature for 20 h. The healed sample exhibited no obvious cracks upon stretching.

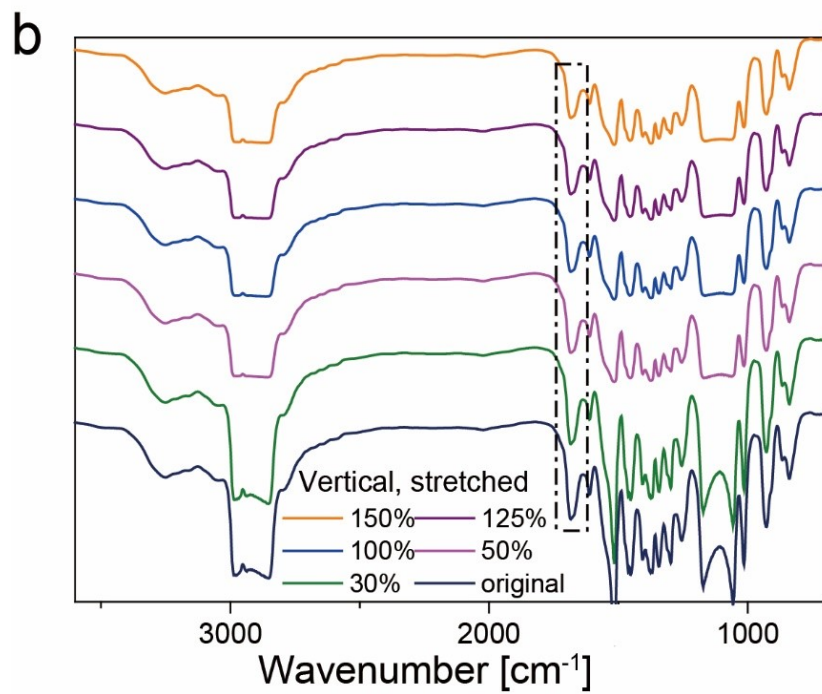
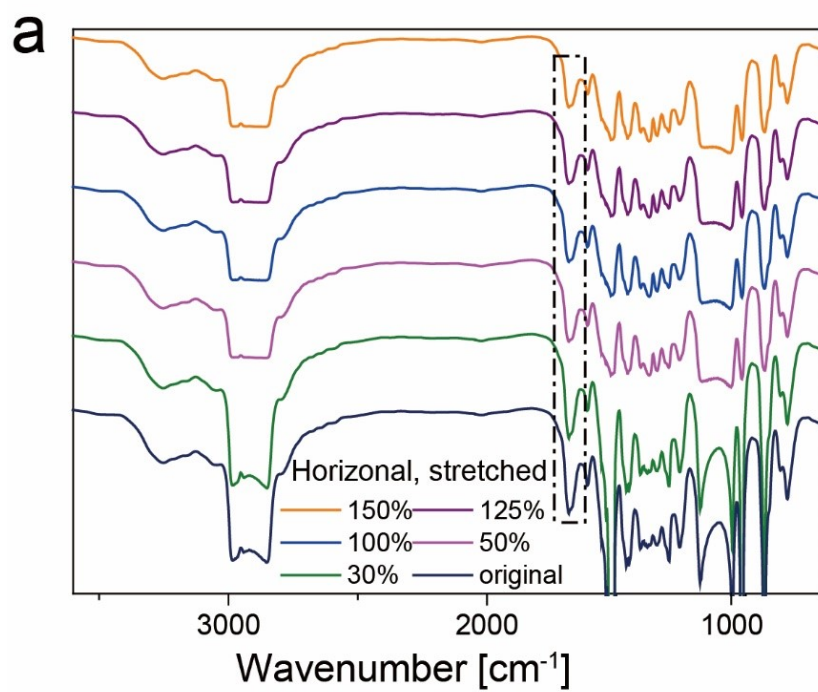




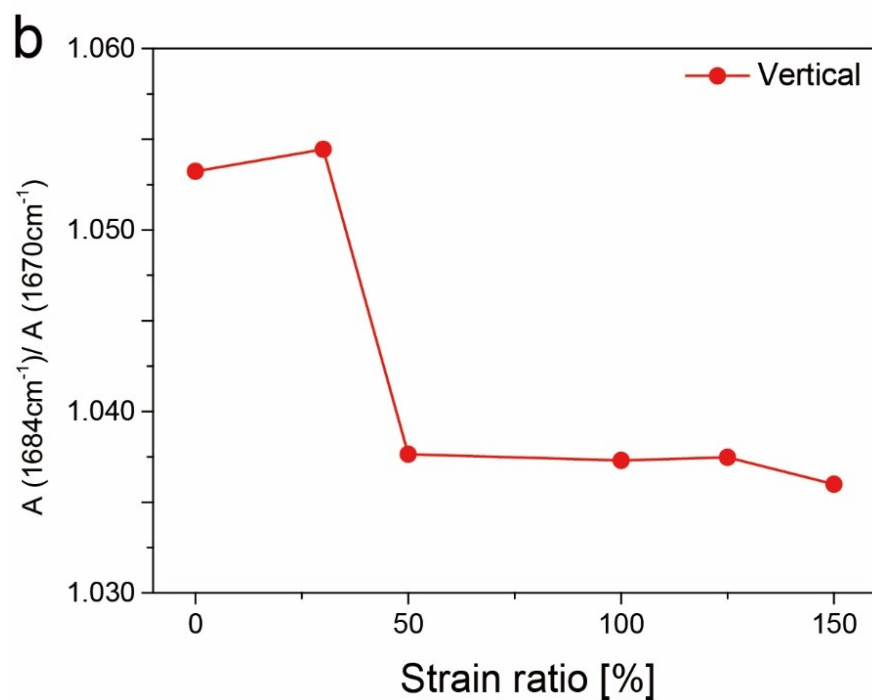
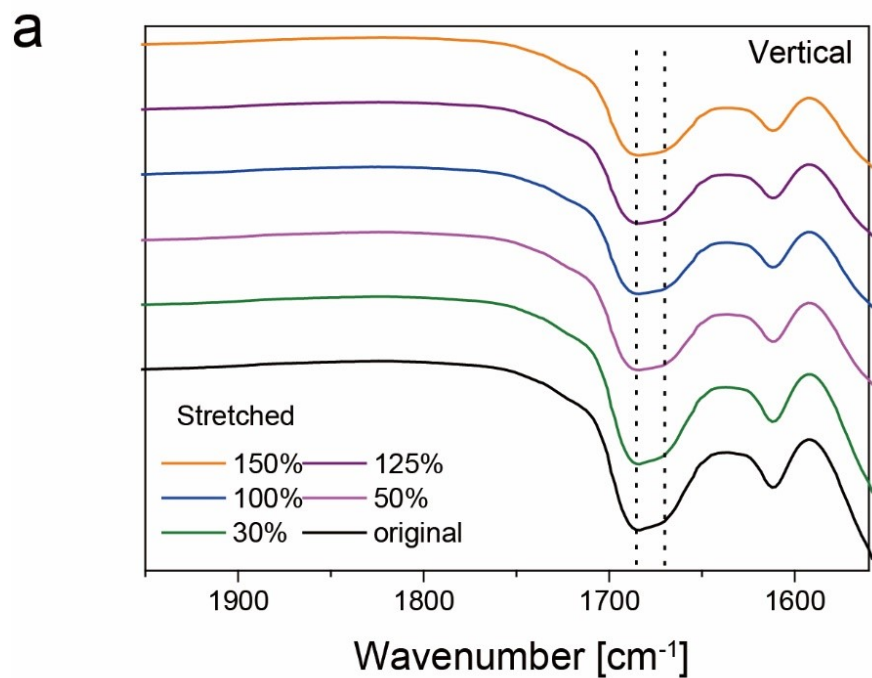
**Figure S18.** Stress–strain curves of the original and healed RC-20-Cu/Mn specimens after healing for 20 h at room temperature.



**Figure S19.** Schematic diagram of polarized FT-IR spectrometry measurement<sup>11</sup>.



**Figure S20.** Polarized FT-IR of RC-1S-Cu in the 3600-700  $\text{cm}^{-1}$ . (a) data for the horizontal polarization. (b) data for the vertical polarization.



**Figure S21.** Analysis of FT-IR for the vertical polarization. (a) Polarized FT-IR spectra of RC-1S-Cu taken during elongation up to 150% strain in the 1950-1550  $\text{cm}^{-1}$ , (b) The value of  $A(1684\text{cm}^{-1})/A(1670\text{cm}^{-1})$  in RC-1S-Cu during stretching up to 150% strain.

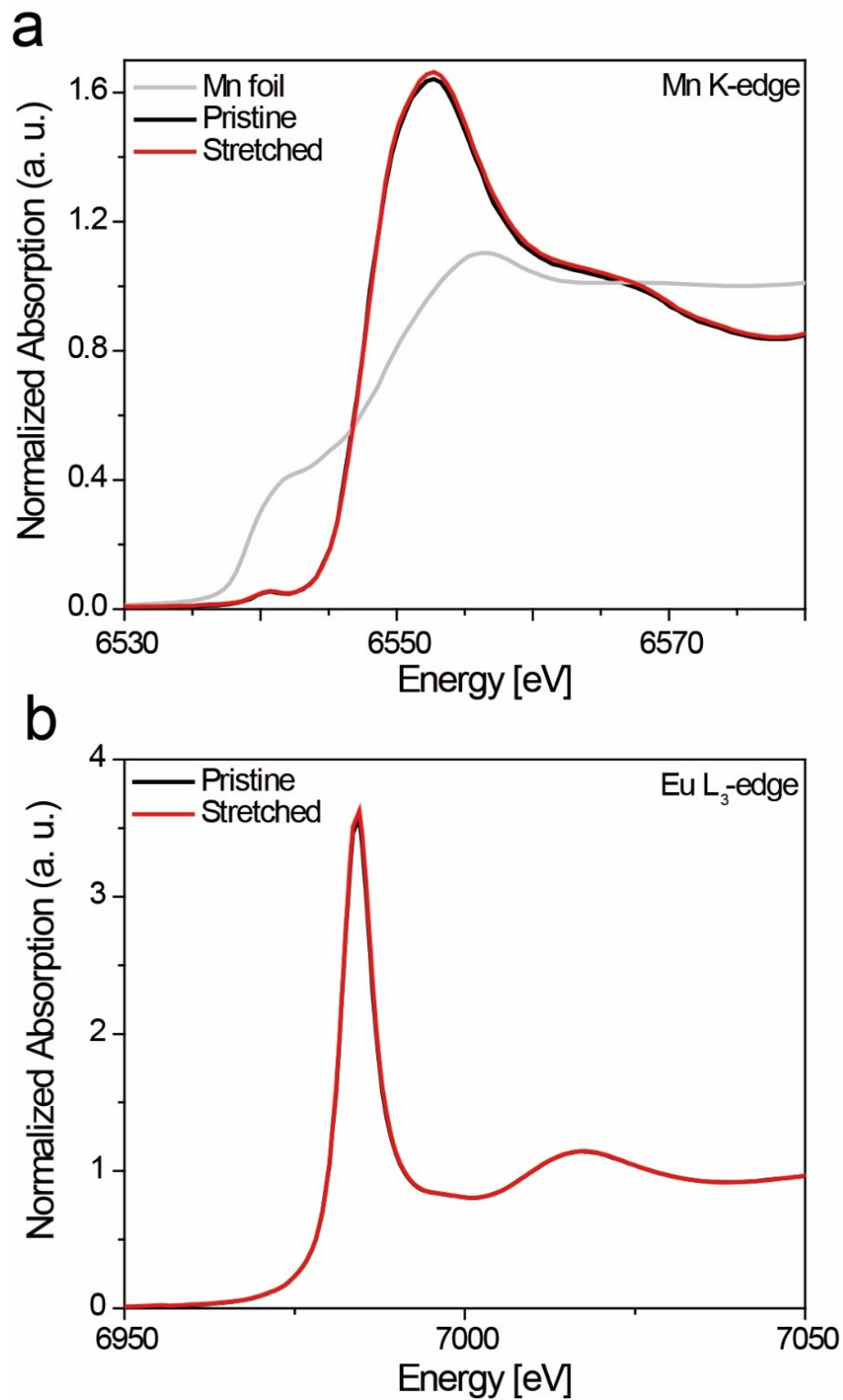


Figure S22. XANES spectra of pristine and stretched samples. (a) data of RC-1S-Mn. (b) data of RC-1S-Eu.

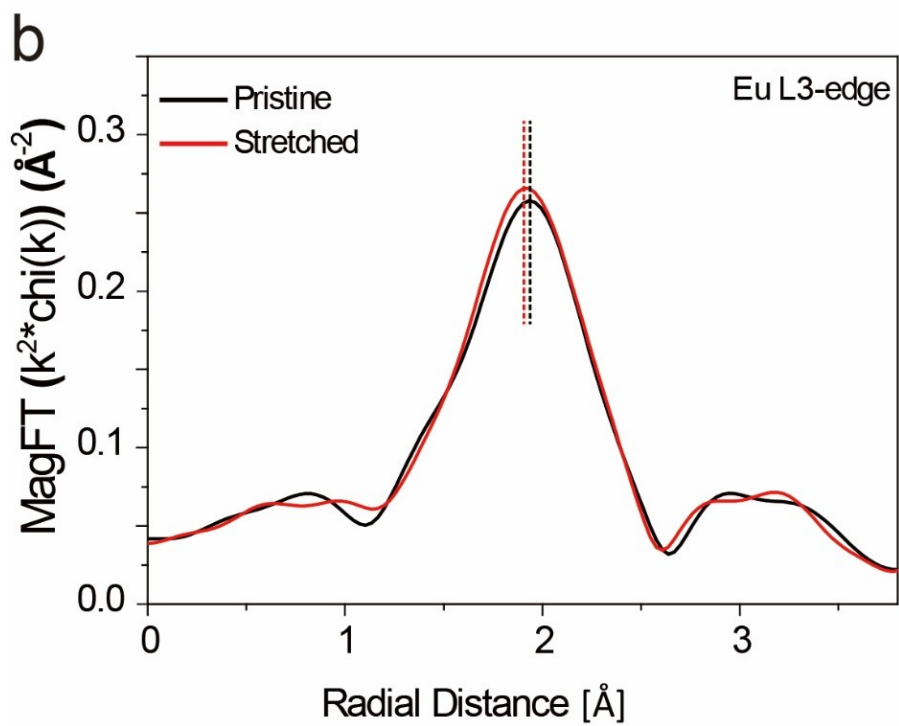
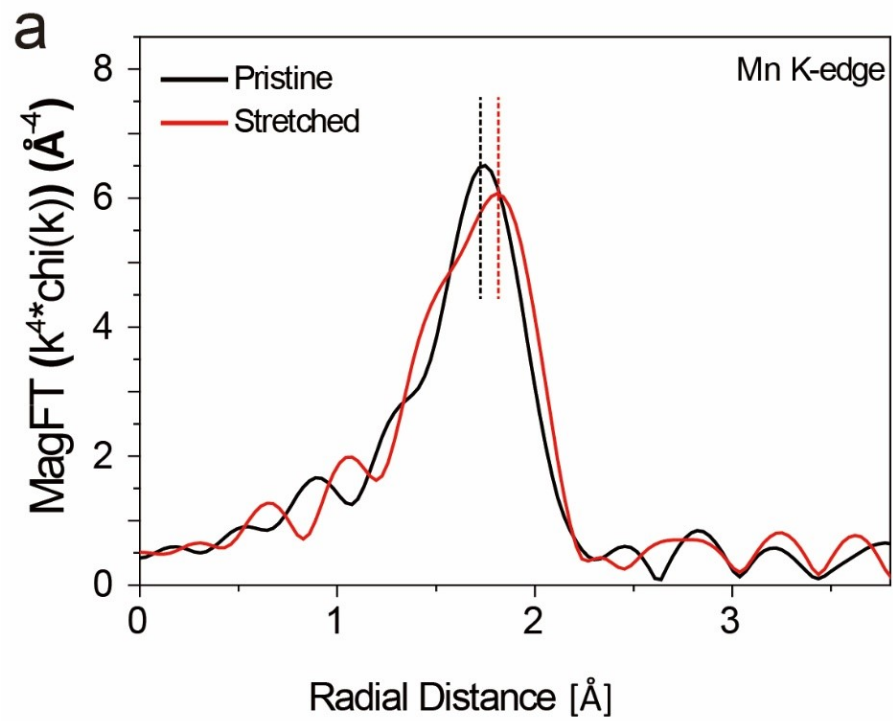


Figure S23. EXAFS spectra of pristine and stretched samples. (a) data of RC-1S-Mn. (b) data of RC-1S-Eu.

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