FOCUS REVIEW

Visualization of Nanomechanical Properties of Polymer Composites Using Atomic Force Microscopy

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Abstract

Atomic force microscopy (AFM) is considered an advanced tool for microscopic study of materials study due to its capabilities for nanoscale spatial characterization. Over the past two decades, the AFM-based nanomechanical characterization technique has been extensively used to investigate the mechanical properties and deformation mechanisms of polymeric materials. This technique enables direct visualization of the micromechanical properties of material surfaces and is referred to as the AFM nanomechanics technique. This review discusses the application of this technique in the study of polymer composites with a specific focus on the significant advances made in tracking the microscopic deformation behavior and visualizing the microscopic stress distributions of materials.

Introduction

Polymer composites play a critical role in today's society, and their excellent mechanical properties and multifunctionality contribute significantly to achieving global carbon neutrality goals, such as the use of new filled rubber in tires. The high strength and multifunctionality of such materials are closely linked to their microstructure, which encompasses multilevel structures such as the aggregated state structure of molecular chains, the interfacial structure of nanocomposites, and the dispersed state. However, conventional testing methods, such as tensile tests and dynamic viscoelastic spectroscopy, can obtain only statistically averaged macroscopic physical properties for polymer composites with complex multi-level micro- and nanostructures [1, 2]. Methods for nano-scale observation of material characteristics, such as transmission electron microscopy (TEM) and scanning electron microscopy (SEM), usually provide information on only the microscopic spatial structure [3, 4]. As a result, it is challenging to establish a direct connection between multi-scale microstructure and macroscopic properties and to accurately understand the microscopic

mechanisms of polymer material properties. Therefore, obtaining information on the multiple physical properties of polymer materials at various micro- and nano-levels and gaining insights into the microscopic mechanisms of their functionality is an essential focus of current materials research.

Recently, the AFM-based nanomechanical technique has emerged as a valuable tool in the fields of polymers, biology, and medicine. This technique allows for the visualization and quantitative characterization of the mechanical properties of materials at the nanoscale [5–7]. By pressing the material surface with a nano-scale tip of a probe using the AFM force mode, the micromechanical properties of the contacted part can be measured, and information at all contact points can be visualized through two-dimensional image processing. This review focuses on the application of the AFM nanomechanical technique to polymer composites by summarizing the work of authors and other researchers as well as the latest developments in the field. In addition, we highlight significant advances in tracking the microscopic deformation behavior of materials and visualizing the microscopic stress distribution, demonstrating the potential of this technique for studying the mechanical properties of polymer composites.

AFM nanomechanics technology

The AFM nanomechanics technique is based on the force model of AFM, in which the force–deformation curve of a tiny contact surface is obtained by pressing the sample

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Fig. 1 (**a**) Schematic diagram of the probe pressing a surface, (**b**) contact between an axisymmetric probe and elastic surface, (**c**) typical force–deformation curve measured in EPDM, and (**d**) elastic modulus image of a PP/EPDM blend
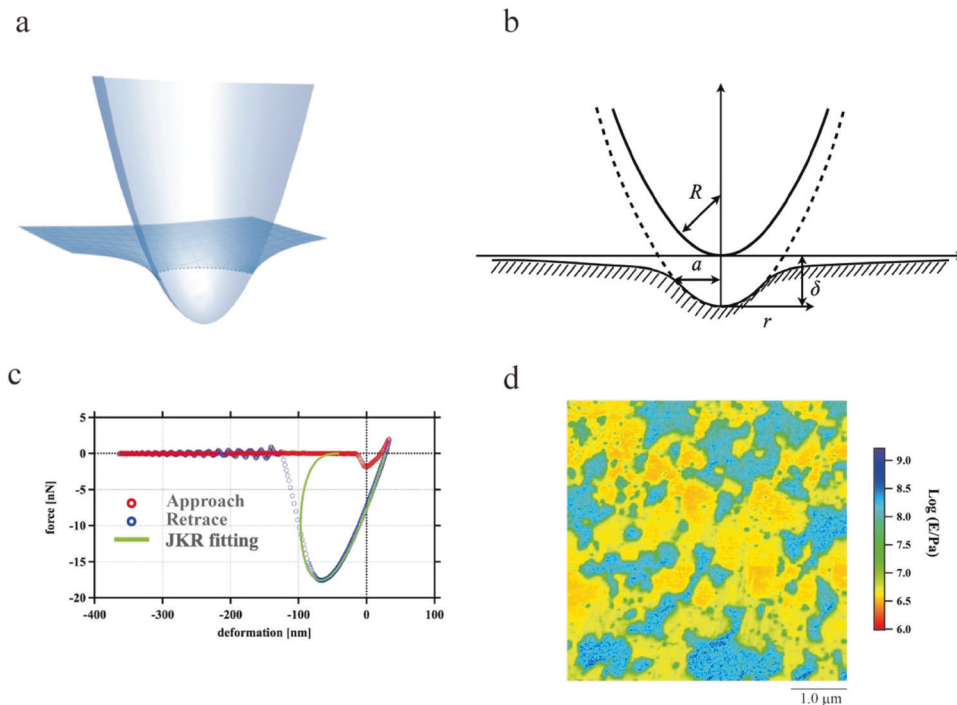
surface with the microscopic probe of an AFM instrument (as shown in Fig. 1a), and the mechanical properties such as elastic modulus and adhesion energy are calculated. The elastomeric contact model of continuum mechanics is used to describe the contact process between the probe and the material and to evaluate the mechanical properties of the material. Commonly used contact model theories are Hertz theory [8], Derjaguin, Muller, Toporov (DMT) theory [9], Johnson–Kendall–Roberts (JKR) theory [10], and so forth. The applicability of contact mechanics models has been discussed in many studies and is very complex, often determined by the mechanical properties of the material, e.g., elasticity modulus and adhesive energy [11]. This is not the focus of this review and is not be discussed in detail here. Polymers, e.g., rubbers and elastomers, are viscoelastic soft materials, and their large adhesion energy affects the contact area between the probe and the material. Thus, JKR theory, including short-range adhesion parameters, is often used in the evaluation of polymeric materials. According to this model, the elastic modulus E and adhesive energy w are expressed by the following two equations [12]:

$$K = \frac{4}{3}\frac{E}{1 - v^2} \tag{1}$$

$$\delta = \frac{a^2}{R} - \left(\frac{8\pi wa}{3K}\right)^{1/2} \tag{2}$$

$$p = \frac{K}{R}a^3 - \left(6\pi wKa^3\right)^{1/2} \tag{3}$$

where v is Poisson's ratio, R is the probe tip radius of curvature, δ is the sample deformation, K is the elastic coefficient, and p is the force, as shown in Fig. 1b. Figure 1c shows a typical force–deformation curve measured in ethylene–propylene–diene monomer rubber (EPDM). By using JKR theory to fit the retrace force curve before the tip leaves the sample, information on the modulus of elasticity and adhesion energy at the contact point can be calculated. A microscopic image of the elastic modulus can be obtained by processing the measured elastic modulus of each contact point in two dimensions. Figure 1d. shows an elastic modulus image of the polypropylene (PP)/EPDM blend. The AFM nanomechanics technique can be applied to obtain important microscopic mechanical properties, such as the nano-elastic modulus, nano-adhesion energy, and nano-deformation, which are widely used in various areas of materials research.

Nanomechanical properties of polymer composites

AFM technology has been applied to the study of polymer composites since its early stages of development. In earlier studies, researchers used conventional cantilever resonance-based tapping mode AFM to investigate the microstructure of polymer composites by differentiating the responses of various components on the phase diagram [13, 14]. Clement et al. were the first to use the lightweight mode to directly observe the micro-dispersed structure of silica in silicone

Fig. 2 (**a**) Elastic modulus mappings for 10 vol % carbon black (CB)/styrene butadiene rubber (SBR) in the original sample; the sample after 250% maximum stretching and recovery from the Mullins effect at 75 °C in vacuum for 3 h, 20 h, and 48 h. The scan size was 1.0 μm. The stretching direction was longitudinal in the elastic modulus mappings. (**b**) Typical stress–strain curves of 10 vol % CB/SBR subjected to cyclic uniaxial tension where the maximum stretch was increased every three cycles. (**c**) Schematic illustration of the microscopic mechanism of the Mullins effect and recovery. Reproduced from Liang et al. [20] with permission from the American Chemical Society
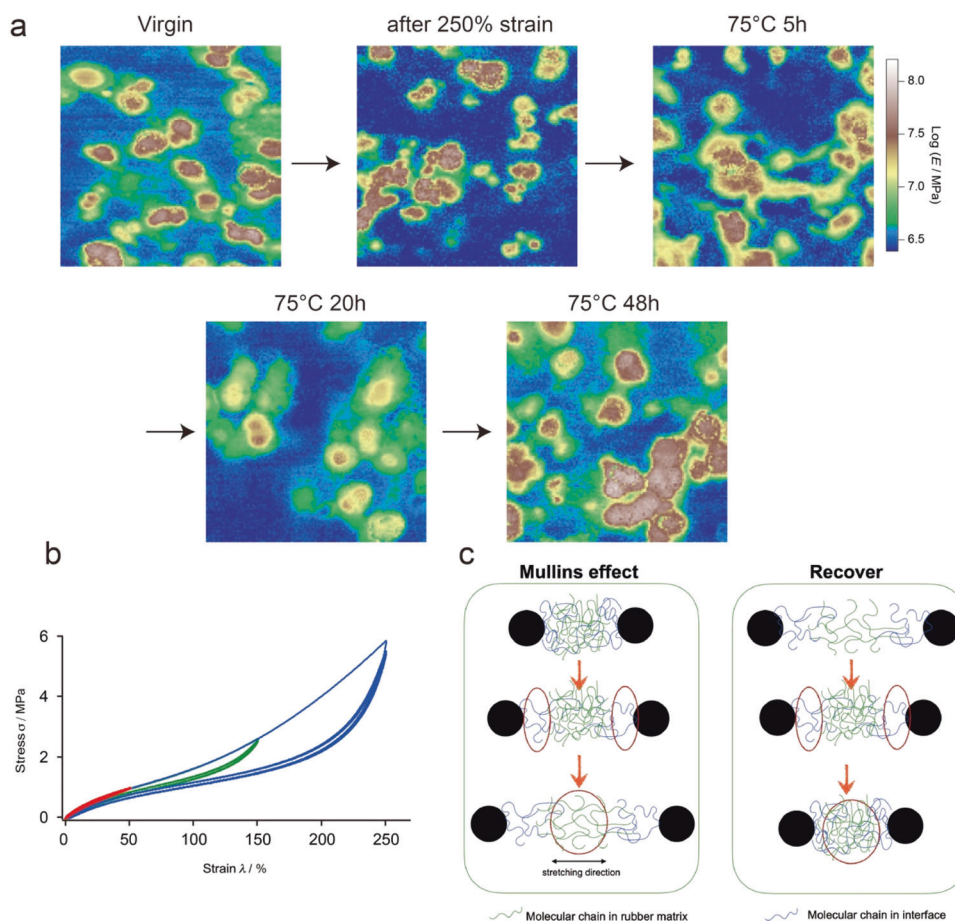
elastomers, which was simpler and more powerful than conventional transmission electron microscopy experiments [15]. With the advancement of AFM nanomechanical techniques, numerous studies have reported on the micromechanical mechanisms of polymers and their composites. For instance, Pakzad et al. observed that poly(acrylic acid)-cellulose nanocrystal/poly(vinyl alcohol) composites have significant deformation, adhesion and modulus gradients between the two phases, which indicate differences in the interface and polymer matrix [16]. Martínez-Tong et al. quantitatively characterized the interphase dimensions and properties of poly(lactic acid)/natural rubber/organo-clay bionanocomposites using peak force quantitative nanomechanics (PF-QNM) to reveal the nanomechanical characteristics of the different matrix samples [7]. In addition, Zhang et al. investigated the elastic modulus map and adhesion force map of poly(lactic acid)/poly(ε-caprolactone)/multiwalled carbon nanotube (MWCNT) nanocomposites using elastic modulus maps and adhesion force maps and found that the introduction of MWCNTs significantly changed the microphase structure of the nanocomposites [17]. The simultaneous grasp of material microstructure and micromechanical properties made an important contribution to the study of the microscopic

mechanisms of polymer composites possessing complex multilevel structures [18, 19].

Recently, we investigated the microscopic mechanism of the Mullins effect in filled rubber using AFM nanomechanics, as shown in Fig. 2 [20]. The Mullins effect is the phenomenon of stress reduction in materials such as rubber and elastomers under cyclic loading (see Fig. 2b). The AFM results revealed that interfacial damage was the dominant factor for the Mullins effect in the low and medium elongation regions, as the stresses were concentrated in the interfacial region. In the high-elongation region, the interfacial damage reached its limit, and the stress was gradually dispersed by the rubber matrix, leading to the release of chain entanglement. Therefore, matrix damage was the dominant factor of the Mullins effect in the high-elongation region. In addition, the recovery from the Mullins effect is similar to the softening mechanism, where the interfacial thickness is recovered first in the initial stage of recovery, followed by the recovery of the elastic modulus of the matrix. In conclusion, the quantitative characterization of micromechanical properties provides essential information for materials research and significantly improves our understanding of the microscopic origin of material mechanical properties.

Fig. 3 (**a**) Elastic modulus mappings of unstretched 13.2 vol % CB/IR, (**b**) elastic modulus mappings of 13.2 vol % CB/IR at a strain of 300%, (**c**) schematic diagram of deformable AFM nanomechanics, (**d**) three-phase separation diagram of Fig. 3b, and (**e**) network structure model of CB-filled rubber

network structure
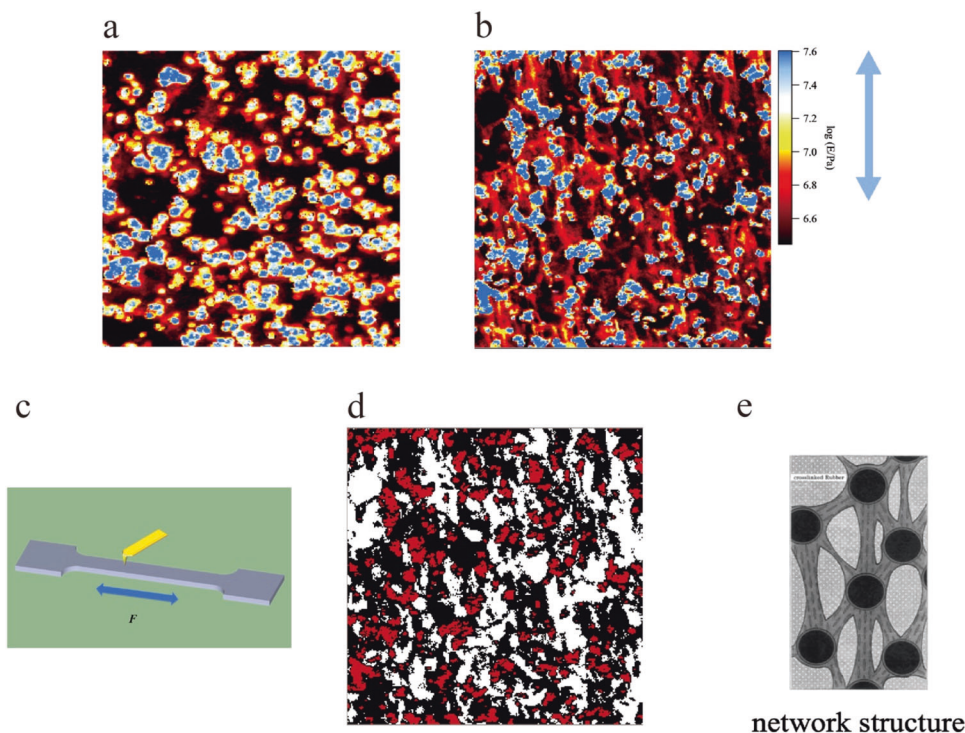
## Visualization of microscopic deformation behavior and nanoscale stress distribution

In the last decade, AFM nanomechanics techniques have become an important tool for research in various material fields. Most studies can evaluate only the elastic modulus and hardness information on the material surface. However, at large deformations, polymeric materials have properties that are different from those of metals and inorganic materials, and their material strength and applications are closely related to their deformation behavior. Even the same polymeric material can exhibit completely different deformation behavior due to differences in the structure of the aggregated state (glassy, rubbery or crystalline state). Therefore, insight into the microstructural changes and nanoscale stress distribution during material deformation has become a critical issue to be addressed in materials research and development. To address this problem, researchers have been exploring ways to track microstructural changes due to deformation. X-ray diffraction methods, such as wide-angle X-ray diffraction (WAXD) and small-angle X-ray scattering (SAXS), have been used to obtain information on average structural changes within the test region [21]. However, these methods have limitations because they cannot provide direct information on the stress distribution inside the material. Recently, with the development of force-responsive luminescent materials, it has become possible to directly visualize the stress distribution inside a material through optical phenomena after applying

a load. This technique allows for the visualization of the state of forces (stress distribution) applied to the material in real space, enabling researchers to understand the progression of structural defects, cracks, and fractures inside the material [22]. However, stress luminescence techniques are limited in their ability to quantitatively assess stresses and are unable to make nanoscale observations.

Therefore, the use of AFM to directly observe the nano-deformation behavior of materials in real space has been highly anticipated. Hiltner et al. used the tapping mode of AFM to directly observe the microstructural changes in polyurethane elastomers during deformation [23]. They found that the initial response to stress is elastic stretching of the soft phase matrix, while yielding and reorganization of the hard phase produce a highly oriented microfiber morphology. Bokobza et al. mounted a small tensile device on an AFM sample holder to enable measurements of deformation [24, 25]. They found that the strain field was highly heterogeneous, depending on the local concentration of filler. Thomas et al. equipped an AFM with an in situ stretching device (designed in their laboratory) to observe the plastic deformation process of semi-crystalline polymers [26]. While this method allowed observation of samples at the micron level, effective in situ tracking at the nanoscale was difficult to achieve due to the noise produced by the stretching device. Early studies of AFM observations of material deformation behavior almost always used the tapping mode to correlate material microstructure and microscopic strain [27, 28]. However, the direct relationship between the microscopic

Fig. 4 In situ elastic modulus mappings of MS-PPO/EPX/CA for (**a**) the unextended state, (**b**) 21% elongation, and (**c**) 65% elongation with a scan size of 5.0 μm. In situ mappings of the matrix region (the black square in Fig. 4a) for (**d**) the unextended state, (**e**) 21% elongation, and (**f**) 65% elongation with a scan size of 1.0 μm. The stretching direction was longitudinal in the JKR elastic modulus mappings. **g** Stretch-induced phase separation (SIPS) effect scheme. Reproduced from Liang et al. [36] with permission from Elsevier
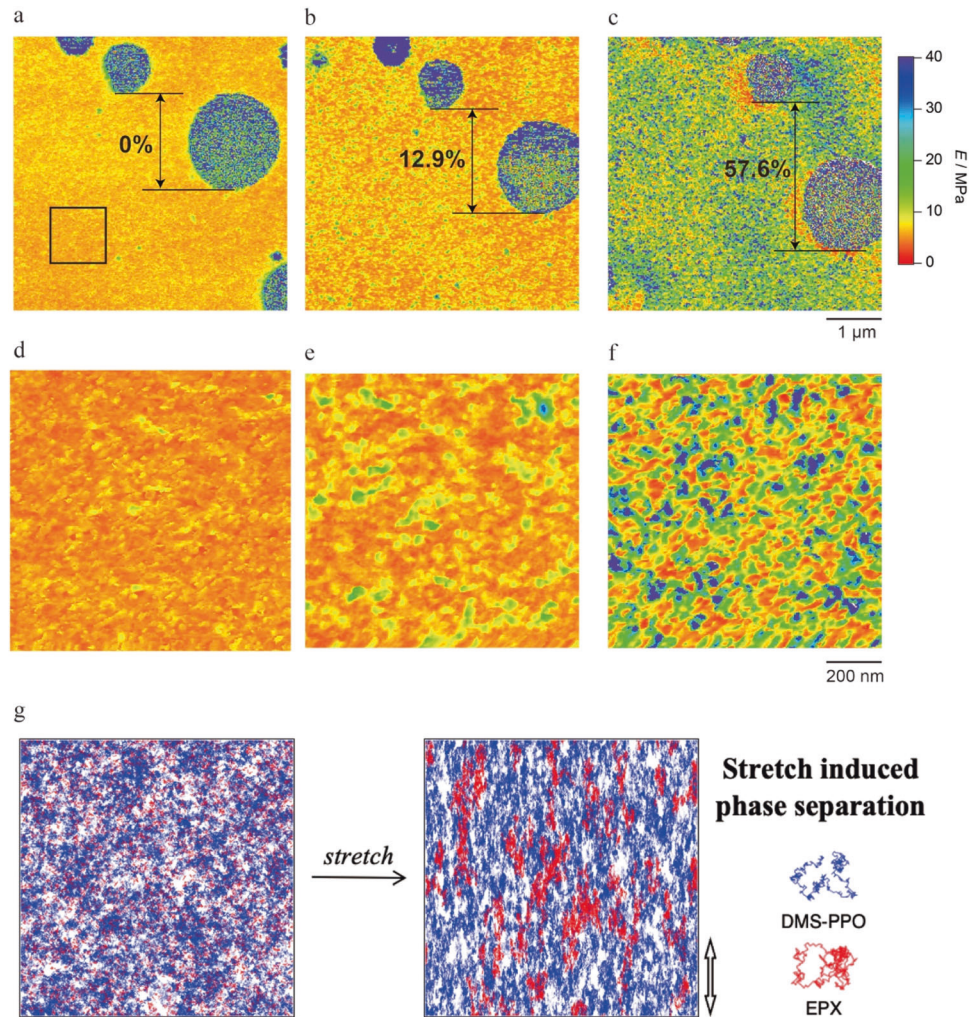
deformation behavior and the macroscopic mechanical properties could not be clarified due to the lack of stress distribution information. Recent advancements in AFM nanomechanics have enabled researchers to investigate the microscopic deformation behavior of materials [29]. By using AFM to measure the mechanical properties of deformed samples, researchers can gain valuable insights into the behavior of materials at the nanoscale. For instance, Morozov used AFM nanomechanics to study the surface morphology and micromechanical properties of filled rubber under tension [30]. The study revealed the emergence of transverse nano-cracks near the point of fracture and demonstrated that the cracking mechanism was affected by the concentration of filler and the distance from the crack tip. These findings provided important information about the behavior of filled rubber under stress. In a study by Wang et al., observations showed that a high-modulus nanofibrous structure formed in unfilled vulcanized IR rubber under high strain, and the authors suggested that this structure was related to strain-induced crystallization (SIC) behavior [31]. Similarly, Zhang et al. reported that vulcanized IR rubber was

divided into a slightly amorphous phase, a highly oriented amorphous phase, and an SIC-associated phase during uni-axial stretching [32]. In addition, a crystalline phase with a "shish-kebab"-like structure was observed. These findings shed light on the complex deformation behavior of vulca-nized IR rubber and highlighted the importance of under-standing the underlying mechanisms of SIC in polymer materials. The authors and colleagues utilized AFM nano-mechanical techniques to visualize the distribution of stress at the nanoscale while tracking microscopic strain behavior [33]. Figure 3 shows the elastic modulus maps of carbon black (CB)-filled isoprene rubber (IR) in both undeformed and deformed states. The elastic modulus in the deformed sample was greater than that in the undeformed sample, and this increase was obviously due to stress, but currently there is no theory that can perfectly explain this phenomenon. However, we consider that the region with increased modulus bore more stress. The stress distribution was clearly non-uniform, as shown in Fig. 3b, with the red regions exhibiting a higher elastic modulus due to increased stress, forming a stress field. The stress fields were interconnected to form a
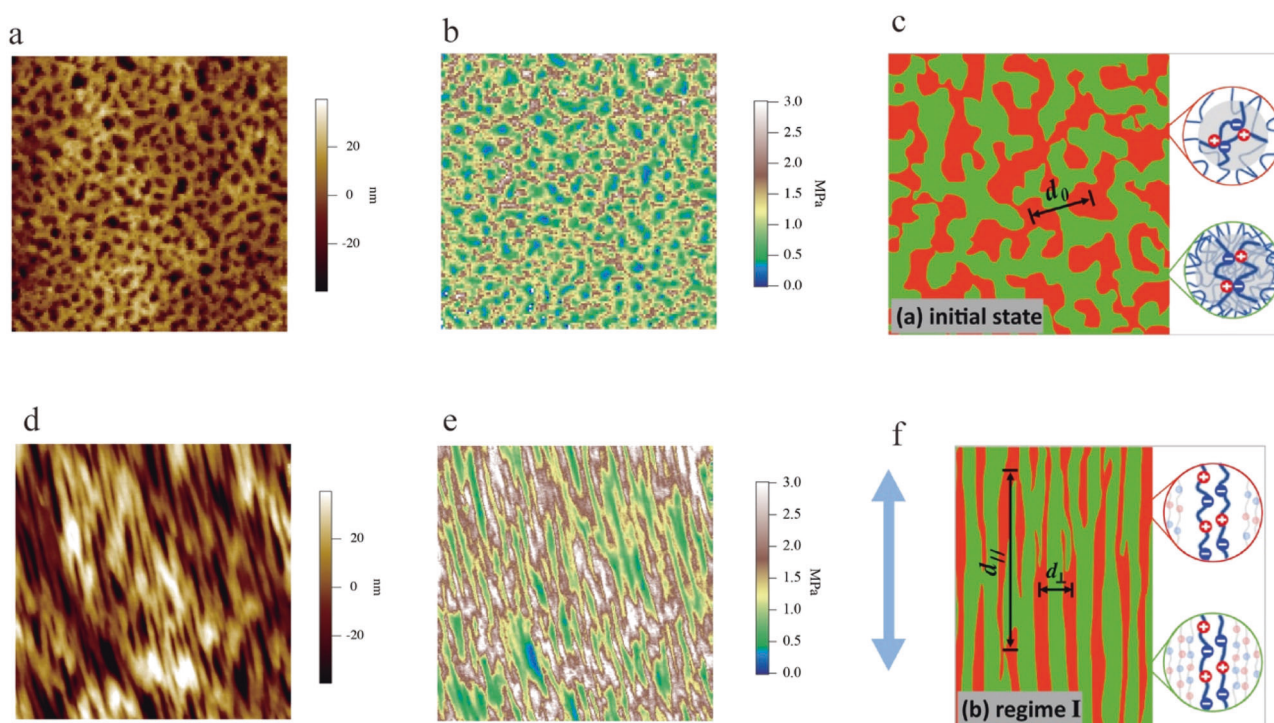
Fig. 5 (**a**) Height image, (**b**) elastic modulus image, (**c**) schematic diagram of the P(Nass-co-MPTC) double-network hydrogel. **d** Height image, (**e**) elastic modulus image, and (**f**) schematic diagram of the deformation mechanism at 200% elongation. P(Nass-co-MPTC) denotes gels composed of the ionic monomers sodium *p*-styrenesulfate (NaSS) and 3-(methacryloylamino)propyl-trimethy-lammonium chloride (MPTC)

stress network structure (see Fig. 3d), which closely resembled the super network structure model proposed by Fukahori et al. [34] (see Fig. 3e). Moreover, the authors proposed a model to predict macroscopic tensile stresses by considering the microscopic stress distribution and spatial structure. These findings provide valuable insights into the relationship between the microscopic and macroscopic behavior of CB-filled IR and can guide the development of new materials with enhanced mechanical properties.

Recently, with the evolution of experimental methods, the authors achieved in situ measurements of the nano-stress distribution during material deformation, i.e., tracing the deformation behavior in the same specific region at different strains [35]. The authors used this approach to investigate the in situ elastic modulus mapping of methoxy silyl-terminated polypropylene oxide (MS-PPO)/epoxy resin (EPX)/silane coupling agent (CA) polymer alloys at different elongation rates, as shown in Fig. 4a–f [36]. The silane coupling agent led to improved compatibility and increased EPX dispersion in the MS-PPO matrix, resulting in a significant increase in the matrix elastic modulus. Interestingly, the stretching of the MS-PPO matrix also induced a stretch-induced phase separation (SIPS) phenomenon, forming aggregated structures tens of nanometers in size, as depicted in Fig. 4g. This SIPS mechanism played a crucial role in toughening the MS-PPO/EPX/CA alloy, as

it reduced the stress on the MS-PPO matrix and acted as a physical cross-linking point, thereby improving the alloy's toughness. By leveraging in situ measurements of the nano-stress distribution, researchers can gain a better understanding of the deformation behavior of materials and ultimately develop more robust materials for a variety of applications. Furthermore, the visualization of nano-stress distribution can be used not only for the study of polymer composites but also to provide important information about biomedical materials, such as hydrogels. Figure 5 shows the nano-elastic modulus map of a hydrogel with a double-network structure [37]. Conventional AFM characterization methods provide only two-dimensional images and make it difficult to obtain the three-dimensional structure of the material, especially to distinguish the double-network structure from the sea-island structure. By visualizing and quantifying the nano-stress distribution, the authors found that both phases of the hydrogel underwent the same deformation behavior and had almost the same stress distribution, which was significantly different from the heterogeneous stress distribution behavior of the sea-island structure. In addition, the microscopic stresses were consistent with the macroscopic stresses when the phases of the nano-stress distribution were substituted into the tandem model for calculation. Therefore, the hydrogel could be considered to have a double-network structure.

Conclusions

AFM nanomechanics techniques can be used to directly characterize important mechanical features such as the elastic modulus and adhesion energy on material surfaces, and they have become a powerful tool for the study of mechanical mechanisms in polymeric materials. In particular, the extension of AFM nanomechanics to materials in deformation allows us to gain insight into the microscopic deformation behavior of materials and even to directly visualize the nano-stress distribution on the sample surface in real space through the change in modulus. This important microscopic information has greatly deepened researchers' understanding of the microscopic mechanisms of materials. In addition, the scale of microscopic information obtained by this technique is close to the level of computer simulations, and it has the potential to serve as a tool to related computer simulations and macroscopic properties of materials. In the future, this technique will not be limited to polymer composites but is expected to be widely used in the fields of biomedical materials and electronic materials.

Compliance with ethical standards

**Conflict of interest** The author declares no competing interests.

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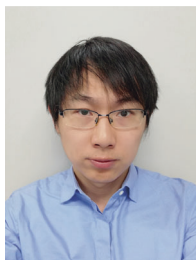
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