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Remote Sensing and Remote Actuation via Silicone–Magnetic Nanorod Composites

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Abstract

The capacity for a soft material to combine remote sensing and remote actuation is highly desirable for many applications in soft robotics and wearable technologies. This work presents a silicone elastomer with a suspension of a small weight fraction of ferromagnetic nickel nanorods, which is capable of both sensing deformation and altering stiffness in the presence of an external magnetic field. Cylinders composed of silicone elastomer and 1% by weight nickel nanorods

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Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Conflict of Interest

The authors declare no conflict of interest.

experience large increases in compressive modulus when exposed to an external magnetic field. Incremental compressions totaling 600 g of force applied to the same silicone–nanorod composites increase the magnetic field strength measured by a Hall effect sensor enabling the material to be used as a soft load cell capable of detecting the rate, duration, and magnitude of force applied. In addition, lattice structures are 3D printed using an ink composed of silicone elastomer and 1% by weight nickel nanorods, which possess the same sensing capacity.

Keywords

3D printing; magnetic elastomers; remote control; soft actuators; soft sensors

The capacity of polymers and polymer composites to respond to stimuli such as heat, light, electrical and magnetic fields, pH, and mechanical stress has led to the development of soft materials that can be programmed to sense environmental cues and respond with controllable and measurable effects.^[1] Of these stimuli, magnetic fields offer remote actuation and remote sensing allowing for specific programmed deformations^[2-4] or enhancement of mechanical properties.^[5] Such materials have potential uses in soft robotics and wearable technology,^[6,7] and in drug delivery and other biological actuation applications due to the remote nature of magnetic fields.^[8,9]

Several research groups have designed materials capable of modulating mechanical properties^[10,11] in response to stimuli, and materials that function as soft sensors.^[6,12] Jackson et al.^[5] designed a lattice of magnetorheological fluid filled struts and demonstrated increased stiffness in response to an applied magnetic field. The authors showed the strength and orientation of the applied field could be used to control mechanical properties. Hellebrekers et al.^[6] created a tactile skin from a silicone elastomer and magnetic microparticle composite, which was able to accurately sense deformation and could be programmed to control electronics based on this capability. However, these materials and other advanced sensing or actuating materials are typically limited to a single dimension, capable of sensing a single stimuli type or actuating in a singular fashion. Therefore, this work aims to expand on the previous developments in magnetic elastomers by fabricating a soft magnetic material that could function as both an actuator and force sensor.

The capacity for a single soft material to combine these functions, such as to sense deformation and respond by altering its mechanical properties, is highly desirable for many applications in soft-robotics and wearable technologies. Indeed, researchers have noted the need for integration of sensing, actuation and computation in soft materials for the advancement of robotics.^[13] This current work, illustrated in Figure 1, presents a silicone elastomer with a suspension of a small weight fraction of ferromagnetic nickel nanowires (Figure 1a,b), which is capable of both altering its stiffness in the presence of an external magnetic field (Figure 1c) and sensing deformation (Figure 1d).

Silicone elastomer, polydimethylsiloxane (PDMS), and nickel nanorod composite precursor were cast in 96-well plates to produce cylinder PDMS–nanorod composites. Longitudinal and transverse nickel nanorod orientation was achieved by applying an external magnetic field during the curing process in the *z*-direction and radial direction of the cylinder PDMS–

nanorod composites, respectively. Longitudinal or transverse orientation of nanorods within the PDMS–nanorod composites (Figure 2a) was confirmed using brightfield microscopy on cross sections of the cured PDMS–nanorod composites (Figure 2b,c), as the nanorods oriented in the direction of the applied magnetic field. Compression dynamic mechanical analysis studies showed that the addition of large nanorods resulted in a higher compressive modulus than obtained through the addition of small nanorods (Figure 2d). When the length of the rods added to the PDMS was increased, a higher Young’s modulus was obtained. The largest enhancement in stiffness occurs when the nanorods are oriented in the same direction as the uniaxial compression (longitudinal orientation) (Figure S4, Supporting Information). This has been previously described by Wang and Weissmuller,^[14] as micropillars and nanowires have been shown to increase the strength of nanocomposite materials. Jackson et al.^[5] also revealed that the uniaxial stiffness of a material is enhanced when the nanorods are oriented in the direction of the compression, but do not have any significant enhancement to the material when oriented transverse to the direction of compression. Thus, the spatial control of the nanorods can be used to program the stiffness of a material in a given axis or create rubber composites with anisotropic characteristics.

The PDMS–nanorod composites were exposed to a magnetic field underneath (Figure S3a, Supporting Information) during compression to analyze the mechanical properties in relation to the strength of the applied field. The magnetic composites were in direct contact with the electromagnet due to the dependent relationship of magnetic field strength and distance from the external magnetic field source. Figure 2d shows that for longitudinal oriented nanorod composites, the application of a magnetic field in-line with the nanorods did not result in a significant change in compressive modulus regardless of the size of the nanorods for all applied fields except the highest applied field. The large rods provided stiffer magnetic composites than the small rods regardless of the strength of the magnetic field applied. However, this was not the case when the magnetic field was oriented perpendicularly to the nanorods, such as for the transverse oriented nanorod composites. When exposed to a magnetic field, the transverse oriented nanorod composites experienced large increases in compressive modulus (Figure 2e). This increase in compressive modulus was related to the strength of the magnetic field applied in a linear manner when exposed to weaker magnetic fields (below 75 G), but signs of saturation were present at higher field strengths. When no magnetic field was applied, magnetic composites with longitudinal oriented nanorods displayed a higher compressive modulus than that of the transverse oriented nanorods composites (Figure S4, Supporting Information).

The characterization of nanorod behavior within the PDMS has been explained by Erb et al.^[10] in which the ferromagnetic nanorods are experiencing a torque in response to the applied magnetic field. A misalignment of the permanent dipole of ferromagnetic nanoparticles in respect to an applied field will yield a higher energy state. The maximum torque that can be applied to the nickel nanorods is then when the nanorods are oriented perpendicular to the magnetic field. This explains the large change in mechanical properties presented by the transverse oriented nanorod composites (Figure 2e). Theoretically, the longitudinal oriented nanorod composite cylinders should not have had any variation in compressive moduli with increased magnetic field, but the marginal increase in compressive moduli observed for the highest applied magnetic field could have been due to the inability to perfectly orient all the

rods at a 90° angle with respect to the applied magnetic field (Figure 2d). Furthermore, the dynamic actuation is reversible given that the transformation of the material is dependent on the magnetic particle response to an applied magnetic field and is consistent with other magnetically actuated silicone based materials.^[2,4,10] This was supported during the PDMS–nanorod actuation studies, as magnetic composite samples were run multiple times across different applied magnetic field levels and there was no noticeable change in the intrinsic compressive modulus of the PDMS–nanorod composites.

Lattice structures of NuSil R40-2181 (NuSil), a heat curable silicone ink, containing 1 wt% nickel nanorods of small (1–4 μm), medium (4–8 μm), and large (8–12 μm) lengths, were 3D printed with strand diameters of ≈0.5 mm (Figure 3a). During the extrusion of the magnetic composite silicone ink, the anisotropic magnetic nanoparticles aligned in response to the shear stress that is formed from the movement of the magnetic ink in respect to the fixed nozzle. A torque is formed due to shear stress changes throughout the volume of the magnetic ink inducing the particles to align with their major axis parallel to the direction of the velocity vector. This leads to longitudinal magnetic particle alignment within the cylindrical magnetic ink strands that are printed.^[15] The alignment of the magnetic particles within the silicone ink was confirmed by imaging 3D-printed lattice structures only two-layer thick (Figure 3b). Rheological characterization and imaging revealed the capacity to print high fidelity 3D structures with this nanorod composite silicone ink (Figure S5, Supporting Information). Similar studies were performed for NuSil–nanorod composites to assess the actuation potential of 3D-printed nanorod structures given the inherent transverse orientation of the nanorods during printing due to shear flow (Figure S5b,c, Supporting Information). There was a significant increase in the compressive modulus of the printed NuSil–nanorod composites when actuated by 400 G versus 0 G (Figure 3c). However, this observed increase was minimal and less than the inherent stiffness variation between magnetic composite samples.

The minimal increase in stiffness of the NuSil–nanorod composites with an applied magnetic field was expected. The 3D-printed nanorod structures consist of overlapping layers that are oriented orthogonal to the tested compression. Therefore, the layers are unable to provide translational forces through the surface of the structure to resist deformation. The layers are also suspended between the overlap of sequential layers which can hinder the continuity of the magnetic composite structures to have a translatable increase in mechanical properties. The intrinsic higher stiffness of NuSil compared to the PDMS used in the current study (Sylgard 184) could also hinder the actuation of the nanorods within the material. Future research should examine whether 3D-printing structures with softer ink compositions, more mechanically robust structures, or with greater control over nanorod orientation could improve the actuation potential of 3D-printed silicone–nanorod composites. These potential developments could be valuable for the advancement of magnetic and sensing technologies given the high-fidelity and dual printing capabilities of the magnetic ink shown in Figure 3d.

Silicone–nanorod composites can function as a load cell where compressive events are sensed remotely by a magnetometer (Figure 4) because the nanorods suspended in the cured rubber are brought in closer proximity to the magnetometer during compression.^[6,7]

Using a triple-axis magnetometer and Arduino microcontroller, PDMS–nanorod composites and 3D-printed NuSil–nanorod composites were subjected to compression, and the change in magnetic field strength was measured to determine if mechanical forces can be sensed remotely. All magnetic composites were in direct contact with the magnetometer during testing. Although sensing with a gap between the composite and the magnetometer is possible, the sensitivity of the signal would decrease with distance from the magnetometer.

Gradually increasing compressive forces in increments of 100 g caused regular changes in the magnetic field strength. The response time of the sensor to the stimuli was essentially instant, limited only by the sampling frequency of the magnetometer. Not only was the magnitude of force remotely detectable by the magnetometer, but also the rate and duration of the force applied (Figure 4a-c). After compressing each structure at 600 g, the compressive force was gradually removed, and the magnetic field strength returned to the baseline at the same rate as the decline in force. The PDMS composite with transverse oriented nanorods displayed a slightly stronger response (43.4 μT at 600 g force) than the composite with longitudinal oriented nanorods (37.6 μT at 600 g force). The 3D-printed NuSil–nanorod composite displayed the strongest response of 50.9 μT at 600 g of compressive force. These results confirm the ability to remotely sense compression events and other mechanical forces in silicone nanorod composites with detection of rate, duration and magnitude of the compressive force. The sensors in the current study sensed forces in the range from 0 to 600 g; however, this range can be altered by using harder or softer elastomers.^[12] For example, a harder elastomer would require greater force to reach the same level of deformation and consequently the same change in magnetic field strength.

Furthermore, cyclic compression testing showed the PDMS–nanorod composites retained their initial magnetic field strengths after many cycles of compression, indicating the potential effectiveness of this remote sensing for biosensing applications. Thirty cycles of uniaxial compression at 300 g of force were conducted on a PDMS–nanorod composite containing transverse oriented nanorods. Each compression cycle was 20 s in duration, and a brief pause was maintained at 300 g of compression force every ten cycles. Figure 4d shows the sensor output matches applied force closely throughout the range of compression and across all 30 cycles. The responses were not reduced over the course of the cyclic compression test and the coefficient of variation of the peak responses for each cycle was 3.2%. These data corroborate previous reports that magnetic-field-based force sensors display repeatability and low hysteresis.^[7]

A silicone composite filled with magnetic nickel nanorods was capable of both remote sensing and remote actuation. Incremental compressions totaling 600 g of force increased the magnetic field strength measured by a Hall effect sensor enabling the material to be used as a soft load cell. In addition, applying a magnetic field perpendicular to the orientation of the nickel nanorods dispersed in PDMS caused an increase in the compressive modulus. Although many studies have used magnets suspended in rubber cushions to sense deformation of the cushion using similar set ups,^[7,16] a few studies have used entirely soft materials.^[6,12] This work presents a material with this remote sensing function and the ability to actuate. A single material capable of sensing and actuation addresses the need for tight integration of smart materials for soft robotics.^[13] For example, the material could be

used for a robotic skin with a large dynamic range of sensing capability to mimic human skin.^[17] In addition, the ability to print discrete domains of magnetic silicone ink in complex geometries opens a multitude of opportunities for remotely sensing and controlling soft materials. Future work will explore the ability of silicone–nanorod composites to sense in three dimensions, as well as determine the location of deformation across its surface, which have been reported in previous materials^[6,7] and to actuate by increasing the compressive modulus to increase the range of sensing capacity.

Experimental Section

Synthesis and Mechanical Testing of PDMS–Nanorod Composites:

Nanorods of varying lengths (4–12 μm), characterized in Figures S1 and S2 (Supporting Information), were incorporated into polydimethylsiloxane (PDMS) (Dow SYLGARD 184 Silicone Elastomer Clear, Dow Chemical Co., Pevely, MO) at 1 wt% concentration with a PDMS base to curing agent ratio of 15:1. The PDMS–nanorod precursor was cured in flat bottom 96-well plates (Corning Incorporated; Corning, NY) at 60 °C for 1 h. A 15 G electromagnet was oriented to yield an external magnetic field in the radial and z -direction, in respect to the cylinder well with the PDMS–nanorod precursor during curing to produce structures with transverse and longitudinal oriented nanorods, respectively.

A cylindrical electromagnet was mounted onto the RSA III DMA and PDMS structures with 1 wt% nanorods were mounted on top of the electromagnet and compressed at a controlled strain rate (0.005 mm s^{-1}). Dynamic mechanical testing was performed on each sample with the electromagnet powered at 0, 1, 1.5, 3, 6, and 12 V. These studies were conducted on PDMS–nanorod structures with small ($N=3$) and large ($N=5$) longitudinal oriented nanorods and with transverse oriented nanorods ($N=3$). The magnetic field strengths produced by the electromagnet at each corresponding voltage were measured with SS49E linear Hall effect sensors (Honeywell; Golden Valley, MN) controlled by an Arduino Mega 2560 microcontroller (Arduino; Somerville, MA). Magnetic field strengths produced by the electromagnet were then reported in Gauss. Compressive moduli (G') data obtained from dynamic mechanical analysis testing were plotted against magnetic field strength values.

Synthesis and Mechanical Testing of NuSil R40-2181–Nanorod Composites:

NuSil R40-2181 (NuSil), a two-part heat curable silicone ink, containing 1 wt% nickel nanorods with lengths of 1–4, 4–8, and 8–12 μm , were 3D printed with an EnvisionTEC Bioplotter (EnvisionTEC; Gladbeck, Germany). Printing parameters (e.g., pressure and plotting speed) were adjusted to yield strand diameters of approximately 0.5 mm. Lattice structures with heights of 4 mm were printed and cured at 160 °C for 4 h. A 3 mm biopsy punch (Miltex GmbH, Rietheim-Weilheim, Germany) was used to form cylindrical magnetic composite samples for uniaxial compression tests. The average compressive modulus was found for structures with transverse oriented long rods ($N=4$) with no magnetic field applied and with an applied magnetic field of 400 G using an ElectroForce (ElectroForce 5500, TA Instruments) at a controlled strain rate (0.001 mm s^{-1}). An ElectroForce was used in place of an RSA III for the usage of a stronger electromagnetic to induce the highest actuation potential (Figure S3b, Supporting Information).

Remote Sensing of Compressive Forces Applied to PDMS–Nanorod Structures:

A MLX90393 triple-axis magnetometer breakout board (SparkFun, Boulder, CO) was wired to an Arduino Mega 2560 microcontroller to communicate via I2C, and the magnetometer board was then mounted onto the bottom geometry platform of the RSA III DMA. PDMS–nanorod composites with 1 wt% nanorods oriented transverse and longitudinal were subsequently placed above the magnetometer sensor, and the RSA III DMA was utilized to exert increasing increments (100–600 g, 100 g increments) of compressive force on the PDMS–nanorod composites. Magnetic field strength data were collected from the output of the Arduino IDE software while the magnetic composites were compressed by the RSA III DMA. The PDMS–nanorod cylinders were compressed from 100 to 500 g of force for at least 5 s at each level up to 500 g of force and for at least 50 s at 600 g of force before returning to baseline. Changes in magnetic field strength from the magnetometer during the tests were expressed in μT , after correcting for the baseline magnetic field strength of the nanorod cylinders under no compression and were plotted against time. Remote sensing of compression events was successfully achieved. The rate, duration, and magnitude of compressive forces were detectable based on the change in magnetic field strength measured by the magnetometer. Additionally, the aforementioned procedure was conducted with blank PDMS cylinders without nanorods to confirm the validity of the results (i.e., lack of interfering external forces) and technique outlined above.

Statistical Analysis:

All results were presented as the mean \pm standard deviation (SD). Statistical analysis was performed by SAS 9.4 Software using one-way analysis of variance (ANOVA) for mechanical testing with and without a magnetic field. Dunnett's post hoc tests were performed to compare the nonexternal magnetic field group against all the applied external magnetic field groups. A blocking factor was placed on the sample groups as each sample was used at every external magnetic field level. Tukey HSD post hoc test was done for multiple comparisons across different nanorod orientation or size groups. Statistical significance was defined by p -values of $*p < 0.05$, $**p < 0.01$, and $***p < 0.001$.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Data Availability Statement

Research data are not shared.

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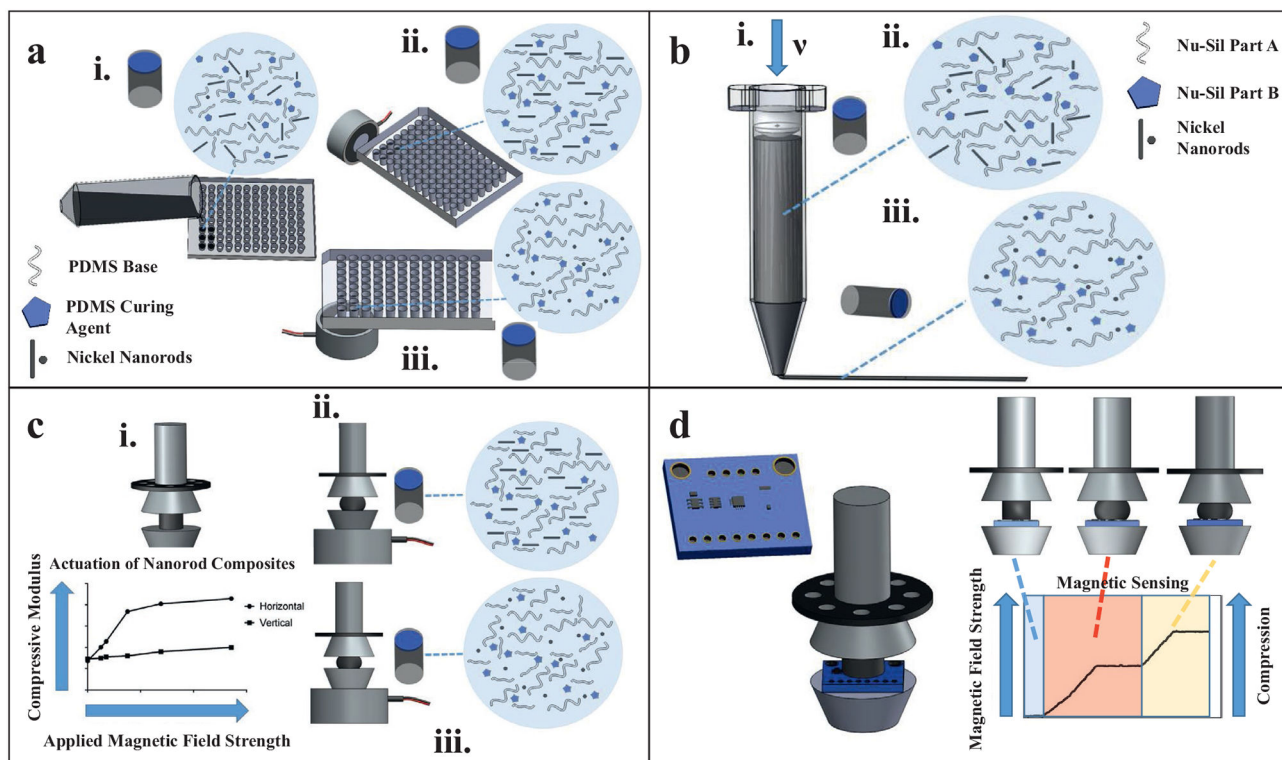


Figure 1.

Schematic of the fabrication process of nanorod composites and its intrinsic remote actuation and sensing capabilities. a,b) The fabrication process for PDMS–nanorod composite structures and control of nanorod orientation. ai) PDMS–nanorod composite cylinders were produced via casting PDMS liquid precursor with nickel nanorods in a 96-well plate. An external electromagnetic field was applied in aii) radial direction and aiii) z-direction of the PDMS–nanorod composite filled wells during the curing process to induce transverse and longitudinal orientation of the rods. b) NuSil R40-2181–nanorod 3D grid structures were created via extrusion-based 3D printing. bi) For extrusion of the magnetic ink, the velocity (v) of the magnetic ink within the nozzle develops a shear stress profile that aligns the anisotropic particles with their major axis parallel to the direction of the velocity of extrusion. Shown is the magnetic particle orientation bii) before and biii) after a velocity profile is developed within the magnetic ink. c,d) The remote actuation and sensing capabilities of the composite structures. ci) The nanorod composite’s orientation-dependent increase in stiffness due to an external magnetic field. cii,iii) The application of an external electromagnetic field during the uniaxial compression of PDMS–nanorod cylinders with different nanorod orientations. Nanorod orientation in the transverse direction cii), in respect to uniaxial deformation, yields an increase in the compressive modulus of the structures when a magnetic field is applied, while longitudinal orientation ciii.) has minimal to no effect. d) The deformation sensing capacity of the nanorod composites. A magnetometer was used to measure the magnetic field strength increase in response to uniaxial compression forces which is caused by the displacement of the nanorods as the composite is deformed.

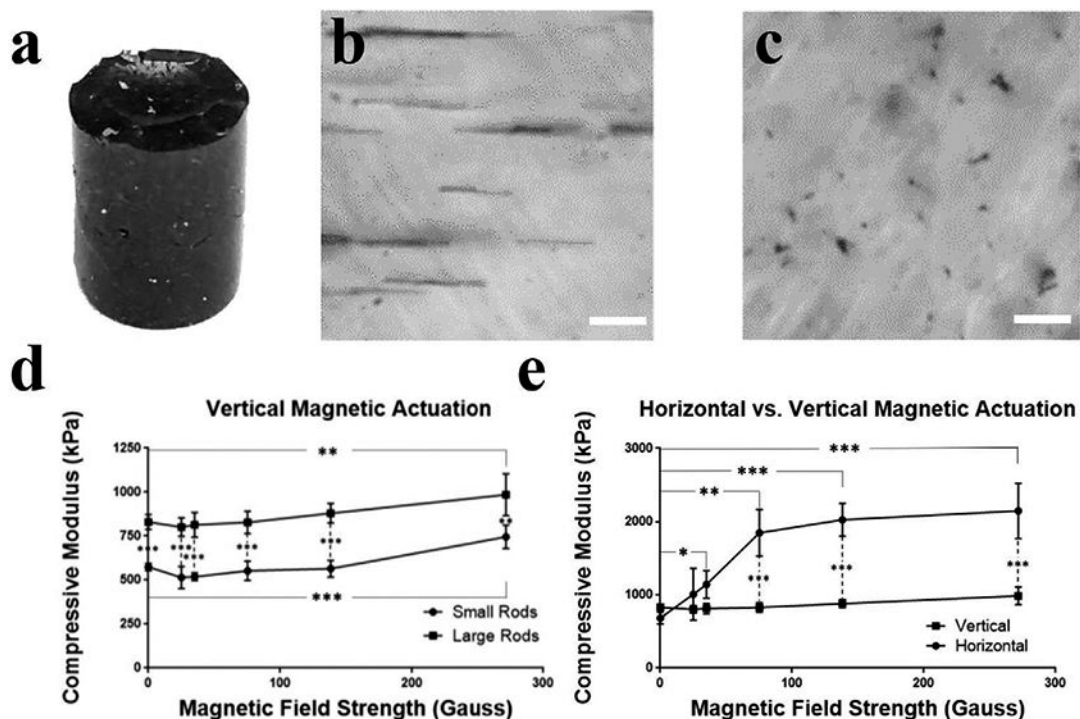


Figure 2. Magnetic-dependent compressive moduli of PDMS structures. a) A casted 1% by weight PMDS–nanorod composite with validation of orientation manipulation via both b) transverse and c) longitudinal orientation. d) 1% by weight longitudinal aligned nickel nanorods of various lengths (small = 1–4 μm ; large = 8–12 μm) and e) 1% by weight longitudinal and transverse aligned large nickel nanorods exposed to an electromagnetic field in increasing intervals up to 272 G. Scale bar = 20 μm .

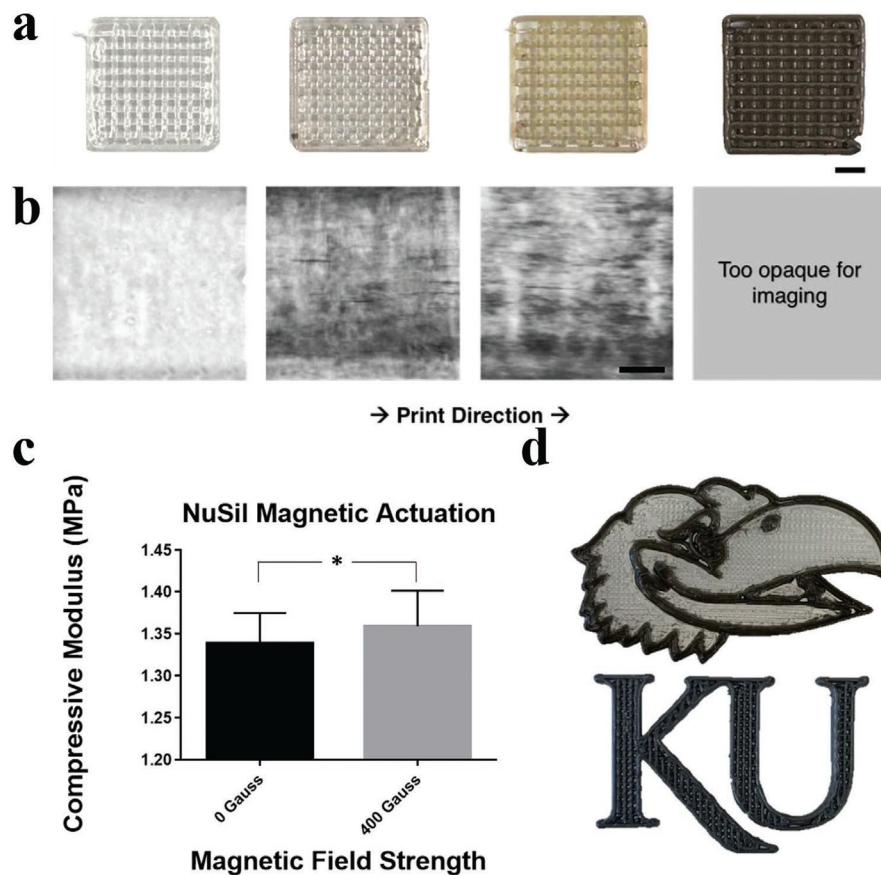


Figure 3. Printability and magnetic-dependent compressive moduli of NuSil R40-2181 structures containing a,b) optical macro- and microscopic images of 3D-printed NuSil containing various concentrations of nickel nanorods (0, 0.1, 1, and 10% by weight). b) Nanorod orientation in the direction of shear flow for NuSil magnetic composites. c) 1% by weight transverse large nickel rod NuSil 3D-printed structures applied to an electromagnetic field of 0 and 400 G. d) The high-fidelity printing capacity for NuSil–nanorod composite structures. Scale bars: macroscopic = 2 mm; microscopic = 50 μ m.

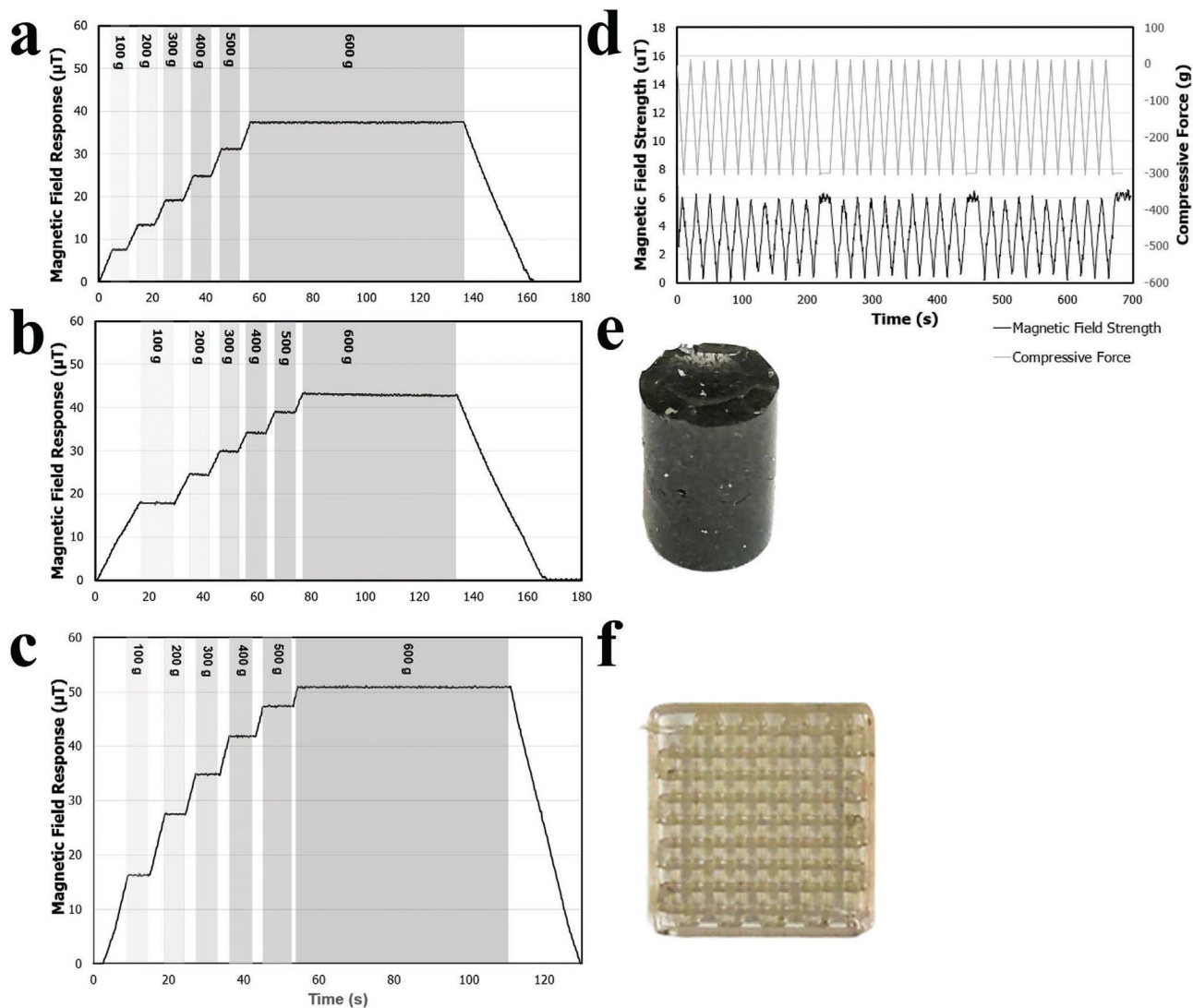


Figure 4. Magnetic field response (μT) plotted against time (s) for compression sensing tests for a) PDMS–nanorod composites with longitudinal oriented rods, b) PDMS–nanorod structures with transverse oriented rods, and c) 3D-printed NuSil R40-2181–nanorod composites. Data are relative to the baseline magnetic field of each composite under no compression. d) An image of the PDMS–nanorod composite structures and e) an image of the 3D-printed NuSil–nanorod composite structures.