Fabrication of magnetostrictive composites using metallic glass as glue

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ABSTRACT

With the continuous expansion of the applications in complex environments, magnetostrictive materials are required to possess not only high performance but also tunable performance. A promising way to achieve this goal is to form composites by combining two or more materials together. However, high performance magnetostrictive composites are still rare, in particular, in a pure metallic form. In this work, we develop a facile and efficient method by using metallic glass as glue, to fabricate pure metallic magnetostrictive composites with high and tunable performance. The Terfenol-D/metallic glass magnetostrictive composites are obtained by a unique thermoplastic forming method under a low temperature (480 K), small stress (114 MPa) and short time (3 min), which exhibit a relatively large field induced magnetostriction (~650 ppm under a magnetic field of 30 kOe) and compressive strength (~185 MPa). Thus, this work provides a promising strategy to design new magnetostrictive materials as well as other metal matrix composites with particular properties.

1. Introduction

The continuous expansion of the applications of materials in complex environments stimulates the increasing demand for materials with particular properties that can be finely regulated, which has thus become a long-sought goal for material scientists [1]. Forming composites by combining two or more materials is a promising way to achieve this goal [2–4]. Crystalline metallic materials generally possess excellent mechanical properties, which enable them as ideal components of composites [5]. However, most of the common binders are thermoplastic polymers by far, such as resins. As a result, it is still hard to obtain composite materials in a complete metallic form. Interestingly, recent work on the thermoplastic properties of metallic glass (MG) exhibits probability to form pure metallic composite materials [6].

MGs, also called amorphous alloys, are a class of metallic materials without long-range ordering of atomic packing, which has superior properties such as high strength and hardness, high elastic limit and excellent corrosion resistance [7–10]. These excellent properties make MGs have broad applications in the fields of sporting goods, medical apparatus and electronic devices [11–16]. Besides these excellent

properties, MGs also possess unique thermodynamic properties, distinct from their crystalline counterpart. Upon heating, MGs generally exhibit an endothermic heat capacity step at the glass transition temperature (T_g), and an exothermic peak at the crystallization temperature (T_x). The temperature range between T_g and T_x is known as the supercooled liquid region (SLR). In this region, the viscosity of MGs drops sharply as the temperature increases [7,12,17–25], which enables the MGs to be thermoplastically deformed like plastics.

It has been found that the thermoplasticity of MGs enables them to serve as binders, which are called "metallic glue" and can combine almost any kind of solid-state materials, such as high entropy alloys (HEAs), ceramics, and oxide glasses (SiO_2) [6]. As a result, such a metallic glue can be used to fabricate bulk composites with certain special properties and excellent mechanical properties [6]. In particular, it is also promising to fabricate bulk materials which are difficult to be obtained by conventional methods, such as magnetostrictive materials.

Magnetostrictive materials are a class of materials that can realize the conversion between electromagnetic energy and mechanical energy and thus are widely used in various transducers, sensors and actuators [26,27]. Among the magnetostrictive materials, the rare-earth alloy

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Tb-Dy-Fe (known as Terfenol-D) has found the most wide applications due to its excellent characteristics, including high magnetostriction, high energy density, high Curie temperature and large magneto-mechanical coupling coefficient [28]. However, the preparation of high-performance Terfenol-D bulk materials is quite difficult. The traditional way to prepare Terfenol-D single crystals and grain-oriented polycrystalline alloys is the directional solidification method [29,30]. This method is very complicated and time-consuming. In order to effectively prepare the bulk magnetostrictive materials, sintering and bonding methods have been developed [31–36]. Different kinds of bulk magnetostrictive composite materials have been obtained by these methods [31–36]. However, some fatal disadvantages are still not overcome, such as long processing time, high processing temperature and poor mechanical properties [35]. As a result, an efficient method to fabricate bulk magnetostrictive materials is still desperately required.

In this work, we explored an efficient and facile route for preparing bulk magnetostrictive composite materials. We used metallic glass (MG) as a "metallic glue" to form bulk Terfenol-D/metallic glass magnetostrictive composites. These composites can be easily fabricated under a convenient condition of low pressure (114 MPa), low temperature (480 K) and short time (3 min). The composites exhibit a relatively large magnetostriction (~650 ppm in a magnetic field of 30 kOe) and good mechanical properties (a compressive strength of 185 MPa). Moreover, the properties of the composites can be finely regulated by modifying the concentration. Thus, the "metallic glue" method is a promising recipe to design new bulk magnetostrictive materials as well as other metal matrix composites with particular properties.

2. Experimental procedure

MG ribbons ($La_{55}Al_{25}Ni_5Cu_{10}Co_5$, abbreviated La-based MG hereafter) were first prepared by a conventional melt spinning process and then cut into small particles by hand. The Terfenol-D ($Tb_{0.3}Dy_{0.7}Fe_2$) powders were purchased in a commercial way. These two kinds of particles were then homogeneously mixed according to the design of the composition. The concentration of Terfenol-D particles varied in a mass fraction range from 20 wt% to 80 wt%. The corresponding volume fraction range is yielded to be 10 vol% to 70 vol% according to the density of Terfenol-D ($\rho_{T\text{-}D}$ = 9.25 g/cm^3) and that of La-Based MG (ρ_{MG} = 6.0 g/cm^3). Zr-based MG ($Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$, namely Vit1) is also used to prepare the Zr-based MG/Terfenol-D bulk composites in the same way.

The mixed particles were placed in the mold and heated in the vacuum chamber to the SLR (around 480 K), and the pressure was applied to 114 MPa and maintained for about 30 s, and then the pressure was rapidly unloaded to 0 MPa, and the specimen was taken out of the chamber and cooled in air. The time of the whole process was less than 3 min.

X-ray diffraction (XRD, RIGAKU miniflex600) with Cu K α radiation was used to detect La-based MG powders, Terfenol-D powders, and the bulk composites obtained by hot-pressing. Differential scanning calorimetry (DSC, Perkin–Elmer DSC-8000) at a heating rate of 20 K/min was used to detect the thermal properties of La-based MG powders. The dimension and characteristics of defects inside the composites were characterized using a three-dimensional computed tomography system (CT, Sanying precision instruments-nano Voxel 3000d, China). Scanning electron microscope (SEM, Fei quanta FEG 450) instrument equipped with energy disperse spectroscopy (EDS) was used to observe the size of powders and the micro morphology and elemental distribution of the composites. Surface quality morphology was observed by confocal laser scanning microscope (CLSM). The magnetostriction was measured by strain gauges. In order to test the magnetic properties of the composites, the magnetic hysteresis loops were obtained at room temperature by a magnetic property measurement system (MPMS3). The densities of the obtained composites were tested by density balance. The hardness and modulus properties were tested by a Hysitron TI 950 nanoindentation

testing system (Bruker, Germany) with a Berkovich tip.

3. Results and discussion

3.1. Main process of the "metallic glue" method

Fig. 1 shows the schematic fabrication process of the designed composite material. As shown in Fig. 1a, material powders of magnetostrictive alloys and amorphous alloys with suitable particle size are mixed completely according to the design of composition ratio, and then put into the mold. It should be noted that the choice of magnetostrictive alloys and amorphous alloys is very flexible, which allows us to achieve various composite materials. Composite materials with different properties can be obtained according to the material design requirement. In this work, we chose commercial Terfenol-D ($Tb_{0.3}Dy_{0.7}Fe_2$) as the magnetostrictive part, and chose La-based MG as the metallic glue due to its wide SLR and good thermoplastic forming ability (TPF). It needs to point out that other MGs are also available to serve as the metallic glue. The mixture then undergoes hot pressing at the SLR of the La-based MG. The plastic-forming ability of metallic glass enables the formation of composite materials very fast. After a short holding time of about 30 s, we can obtain the final bulk composite materials. As a result, the new magnetostrictive composites can be easily fabricated by this method.

The X-ray patterns of Terfenol-D and La-based MG powders are shown in Fig. 1b. As shown in the XRD spectrum of Terfenol-D powders, all the peaks well match the standard spectrum of the Terfenol-D alloy. Meanwhile, La-based MG powders exhibit a broad halo diffraction peak in the range of 25–40°, which is the typical structure characteristic of an amorphous solids. In order to further determine the thermoplastic forming temperature of La-based MG powders, the DSC curve of La-based MG is shown in Fig. 1c. The glass transition temperature (T_g) of La-based MGs is found at 456 K, the crystallization point (T_x) is determined to be 514 K. Therefore, this La-based MG possesses a wide SLR around 60 K ($\Delta T = T_x$ - T_g), which enables us to choose the hot-pressing temperature at 480 K. Fig. 1d shows the SEM image of La-based MG powders. The size of La-based MG powders is homogeneous, which is mainly in the range of 50–250 μm, and the average particle size is around 150 μm, as shown in Fig. 1e. On the other hand, the size of Terfenol-D particles shown in Fig. 1f is less homogeneous, exhibiting a wider size range of 20–370 μm, and the average particle size of Terfenol-D powders is 110 μm. Wide particle size distribution is favorable to the formation of bulk composite materials, since fine particles can fill in the pores between large particles, and reduce the porosity of the material.

3.2. Microstructure of bulk composites

According to the hot-pressing method described in Fig. 1a, we successfully fabricated full-metallic MG/Terfenol-D bulk composites, as shown in the inset of Fig. 2a. It is noted that the required condition is convenient to achieve, including a low pressure (114 MPa), low temperature (480 K) and short time (3 min). The XRD spectrum of the Terfenol-D/MG bulk composite is displayed in Fig. 2a. A typical amorphous hump is observed around 30°, corresponding to the La-based MG, and the characteristic peaks of Terfenol-D can also be clearly seen. No extra peaks are observed. This result indicates that the two phases coexist in the composite, and the MGs matrix remains amorphous after the hot-pressing process.

In order to further study the bonding performance of the bulk composites, the surface image and the element distribution of the composite with 40 vol% Terfenol-D is shown in Fig. 2b. There is a clear element separation observed at the interface, and it is easy to determine the bright area B as the Terfenol-D particles and the dark area A as the La-based MG matrix respectively. This result indicates the diffusion of element atoms does not take place. Fig. 2c-d further exhibit the interface structure of the bulk composites in a larger area. No obvious cracks are found around the interface between two phases, indicating that the
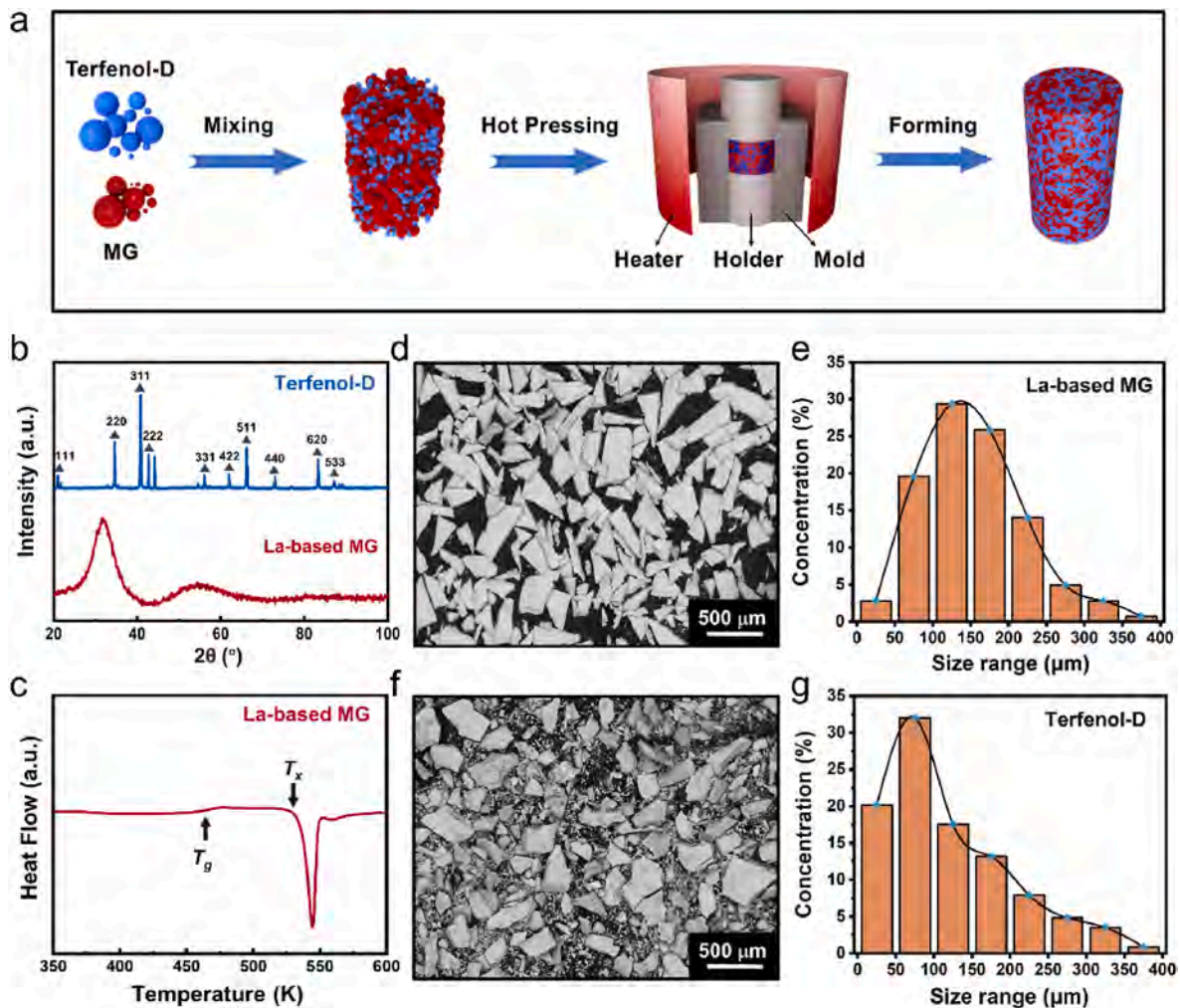
Fig. 1. Process flow chart and material characterization. (a) A schematic diagram of hot-pressing. (b) XRD patterns of Terfenol-D and La-based MG. (c) DSC curves of La-based MG. (d) SEM image of La-based MG powders. (e) Particle size distribution of La-based MG powders. (f) SEM image of Terfenol-D powders. (g) Particle size distribution of Terfenol-D powders.

combination of the two phases is good. The fracture morphology is shown in Fig. 2e–g where severely deformed areas can be clearly seen (illustrated by the red arrows). Surface defects such as pits and holes are seldom found in these severely deformed areas, which means that La-based MG matrix tightly surrounds the Terfenol-D particles. Thus, MG acts as the glue to bond the Terfenol-D particles tightly.

The CT system is also used to observe the inside structure of the composite material from the top view. Taking the middle position as the reference, two photos were taken at each interval of about 200 μm upward and downward, and a total of five photos were taken, as shown in Fig. 2c. The blue part of the image is La-based MG, while the white part corresponds to Terfenol-D. It can be clearly seen that the two materials uniformly distribute inside the sample.

3.3. Magnetic performance

Fig. 3a shows the magnetostriction curves of the composites containing between 10 vol% and 65 vol% Terfenol-D along the direction of the applied magnetic field. The applied field is up to 30 kOe. The inset represents the magnetostriction curve of the original Terfenol-D sample, which exhibits a saturation magnetostriction of around 1100 ppm. The magnetostriction of the composites at 30 kOe is summarized in Fig. 3b. With the increase of the Terfenol-D concentration, the magnetostriction shows a tendency that first rises and then declines, reaching a maximum of the magnetostriction (650 ppm) at the 55 vol% Terfenol-D. Therefore,

the magnetostriction of bulk composites does not monotonously increase with the increase of the Terfenol-D concentration, leaving an optimal ratio exhibiting the highest field induced magnetostriction.

It is well known that the magnetostriction of Terfenol-D can be dramatically improved under the application of uniaxial compressive stress [33,37]. In order to further improve the performance of bulk magnetostrictive composites, the magnetostriction of the optimal ratio (55 vol% Terfenol-D) is measured under a compressive preload and shown in Fig. 3c. It is found the magnetostriction at 30 kOe increase from 630 ppm to 700 ppm when the prestress increases from 0 MPa to 18 MPa.

The magnetization of bulk composites with different contents is shown in Fig. 3d. The saturation magnetization (M_s) of Terfenol-D is 97 emu/g, while that of the La based MG is almost 0 emu/g (the curve is not shown here), indicating it is a nonmagnetic material. The saturation magnetization of bulk composites is summarized in the inset of Fig. 3d. It is found that the magnetization of composites monotonously increases from 14 emu/g to 73 emu/g as the concentration of Terfenol-D increases from 10 vol% to 65 vol%. Consequently, the magnetization can be finely regulated by the design of the composition.

3.4. Mechanical performance

The mechanical performance is also of significant importance to the potential applications of these bulk composite materials, so we
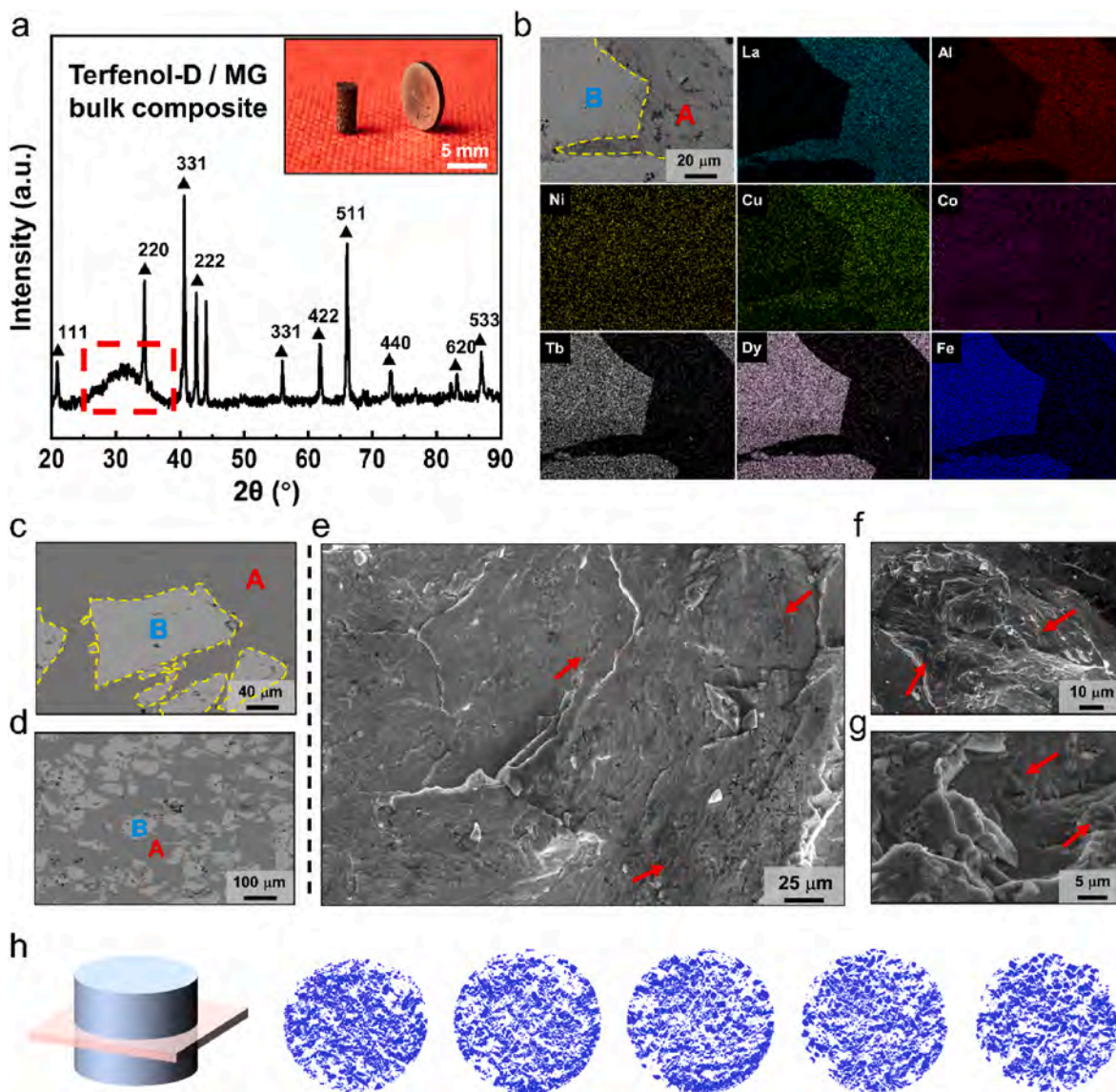
Fig. 2. The microstructure of the bulk composite with 40 vol% (i.e., 50 wt%) Terfenol-D. (a) XRD pattern of the composite. The inset exhibits the image of obtained bulk composites. (b–d) Surface SEM and EDS images of the composite. The yellow dashed line represents the interface between the two phases. A represents the La-Based MG and B represents the Terfenol-D particles. (e–g) Fracture morphology of the composite. The red arrows are used to label the severely deformed areas. (h) Cross-sectional CT images at the center positions. The illustration shows how the sample is scanned in CT.

measured the mechanical behaviors of the bulk composites from several aspects, including hardness, modulus, and stress-strain curves, as shown in Fig. 4. The hardness and modulus of the bulk composites are shown in Fig. 4a and b, respectively. In order to investigate the influence of MG matrix on the mechanical performance, we also fabricated the Zr-based MG/Terfenol-D bulk composites for comparison. Both of the hardness and modulus of the La-based MG composites exhibit an inverse V-shaped change tendency: the property increases first and then decreases with the increase of the Terfenol-D concentration. As a result, with La-Based MG as the matrix, a maximum value is achieved at the 55 vol% Terfenol-D where the hardness maximum is 365 HV and the modulus maximum is 48 GPa. The Zr-based MG/Terfenol-D bulk composites exhibit the same inverse V type tendency, while the hardness maximum is 668 HV and the modulus maximum is 106 GPa at the optimal ratio of 50 vol% Terfenol-D. The higher property values with Zr-based MG as the matrix mean that the mechanical properties of bulk composites are mainly determined by the MG matrix.

The compressive stress-strain curves of the bulk composites with different fraction of Terfenol-D at room temperature are shown in

Fig. 4c. It is found that the compressive strength of the composite is 502 MPa at 10 vol% Terfenol-D, and it decreases gradually with the increase of Terfenol-D content. As the Terfenol-D content increases to 70 vol%, the compressive strength decreases to only 75 MPa. The result of a Zr-based MG composite with 40 vol% Terfenol-D is also shown in Fig. 4c, which exhibit a large compressive strength of 420 MPa, much higher than that of La-based MG composite with the same volume fraction of Terfenol-D. This result further reveals that the MG matrix plays a dominant role in the mechanical properties of the bulk composites.

3.5. Mechanism of the existence of an optimal ratio

In principle, the property should monotonously increase with the increase of the Terfenol-D content. However, according to Figs. 3–4, there is an optimal ratio exhibiting the best comprehensive properties in the MG/Terfenol-D bulk composites. Thus, in order to further improve the properties of bulk composites, it is of importance to unravel the underlying reason for the existence of the optimal ratio.

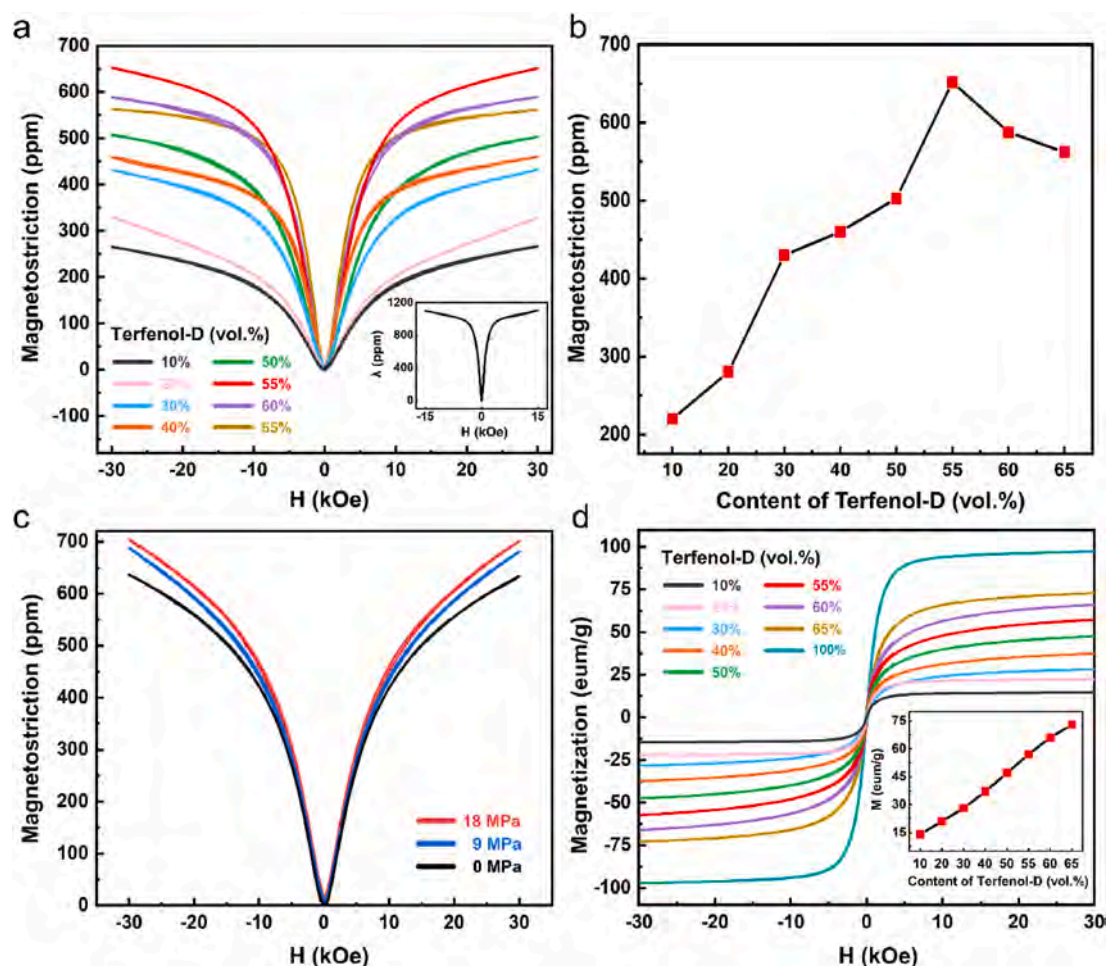The key reason for the existence of the optimal ratio is tightly related

Fig. 3. Magnetostriction and magnetization properties of composite materials. (a) Magnetostrictive curves of La-based MG\Terfenol-D bulk composites in different ratio. The inset image representing the magnetostrictive curve of Terfenol-D. (b) Magnetostriction change with different proportions of composites under a magnetic field of 30 kOe. (c) Magnetostriction curves of the bulk composite with 55 vol% of Terfenol-D under compressive prestress. (d) The magnetic hysteresis loops of composites with different ratio. The inset image represents the saturation magnetization change with the concentration of composites.

to the fact that the low fraction of MG may lead to a low quality of the composite sample. When the MG concentration is too low, the MG particles cannot effectively bond all the Terfenol-D particles, leaving a large number of pores in the composite, and thus the obtained bulk composites are in a low quality. The decrease of the sample quality can be reflected by the relative density change, as shown in Fig. 4d. The density of Terfenol-D is 9.25 g/cm^3, while the density of La-based MG is 6 g/cm^3. Therefore, we can estimate the theoretical density of each composite. We then measured the density of each bulk composite and calculated the relative density accordingly. As shown in Fig. 4d, the relative density of composites decreases gradually with the increase of the Terfenol-D concentration. When the relative density is too low, the properties of bulk composites will sharply decrease accordingly.

To further exhibit the low quality of bulk composites with the high fraction of Terfenol-D, we investigate the surface microstructure of the composite with 70 vol% (i.e., 80 wt%) Terfenol-D. Fig. 5a–c shows the progressively enlarged SEM images of the surface of this bulk composite, from which we can see that the surface quality of the sample is very low, exhibiting many surface defects such as pits and holes. Fig. 5d–f shows the SEM photos of the fracture morphology of this composite. Many particles are exposed in these photos, indicating that the bulk composite is not well bonded due to the low concentration of metallic glass particles. In order to show the sample quality in a clearer way, we also provide the surface photograph of the composites with Terfenol-D fraction of 10 vol%, 40 vol% and 70 vol% by using the confocal laser

scanning microscope (CLSM), as shown in Fig. 5g–i. The surface of the samples with low Terfenol-D content is globally smooth and flat, while the surface quality gradually decreases with the increase of Terfenol-D content. This is another solid evidence to reveal that the sample quality is very low for the high Terfenol-D compositions. Therefore, the magnetostriction and mechanical strength of bulk composites do not monotonously increase with the increase of the Terfenol-D concentration and show the highest value at an optimal composition, which is consistent with the previous studies [32,33].

3.6. Characteristics of the "metallic glue" method

The new bulk composites and the related "metallic glue" method exhibit many advantages. Table 1 summarizes the properties and fabrication method parameters of different bulk magnetostrictive composite materials. According to Table 1, the compressive strength of the MG/Terfenol-D composites in this work is better than the results of other magnetostrictive composites. As shown in Fig. 4, the matrix plays a dominant role in the mechanical properties of the bulk composites, so the high compressive strength of MG/Terfenol-D composites is mainly attributed to the high strength of the metallic glass matrix, which serves as the metallic glue to combine the particles in the composite. When the polymers serve as the glue part, the obtained bulk composites exhibit a very large magnetostriction, but the compressive strength is generally low [31–33]. Meanwhile, the required temperature is generally low, but
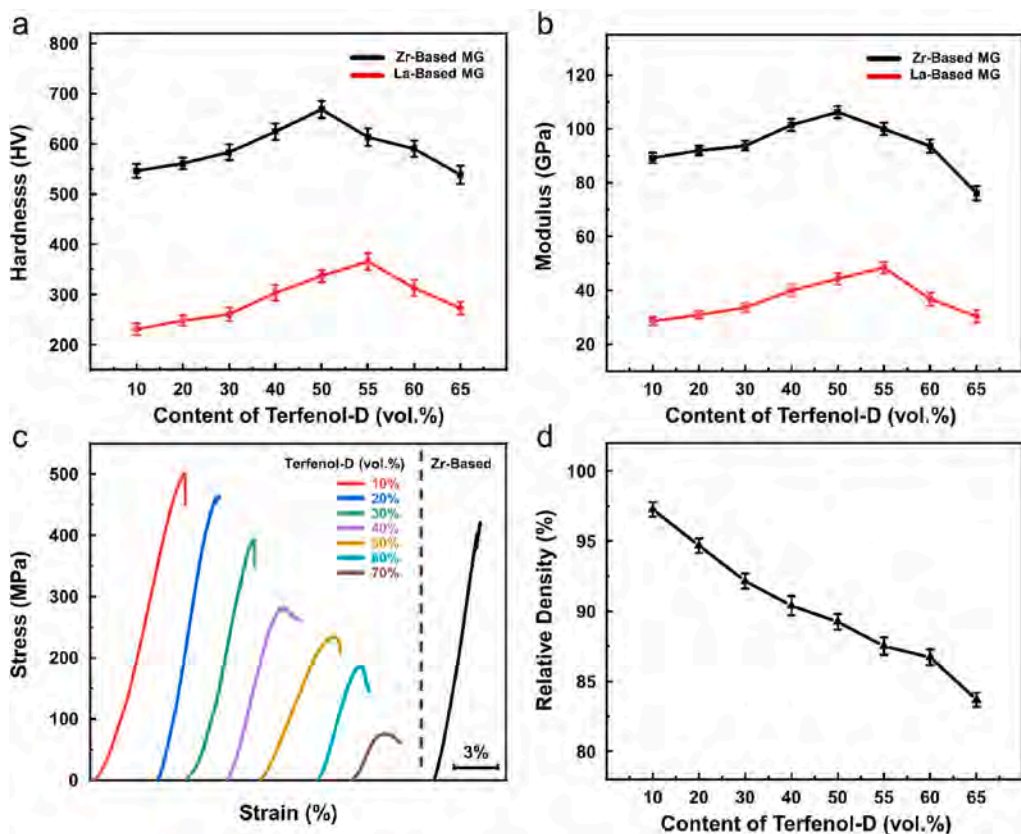
Fig. 4. Mechanical properties of bulk composite materials. (a) Hardness curves of bulk composites with La-Based MG and Zr-Based MG as matrix at different ratios. (b) Modulus curves of bulk composites with La-Based MG and Zr-Based MG as matrix at different ratios. (c) (The left) Stress-strain curves of Terfenol-D/La-based MG bulk composites with 10 vol% to 70 vol% Terfenol-D. (The right) Stress-strain curves of Terfenol-D/Zr-based MG bulk composites with 40 vol% (i.e., 50 wt%) Terfenol-D. (d) Relative density change in Terfenol-D/La-based MG bulk composites.
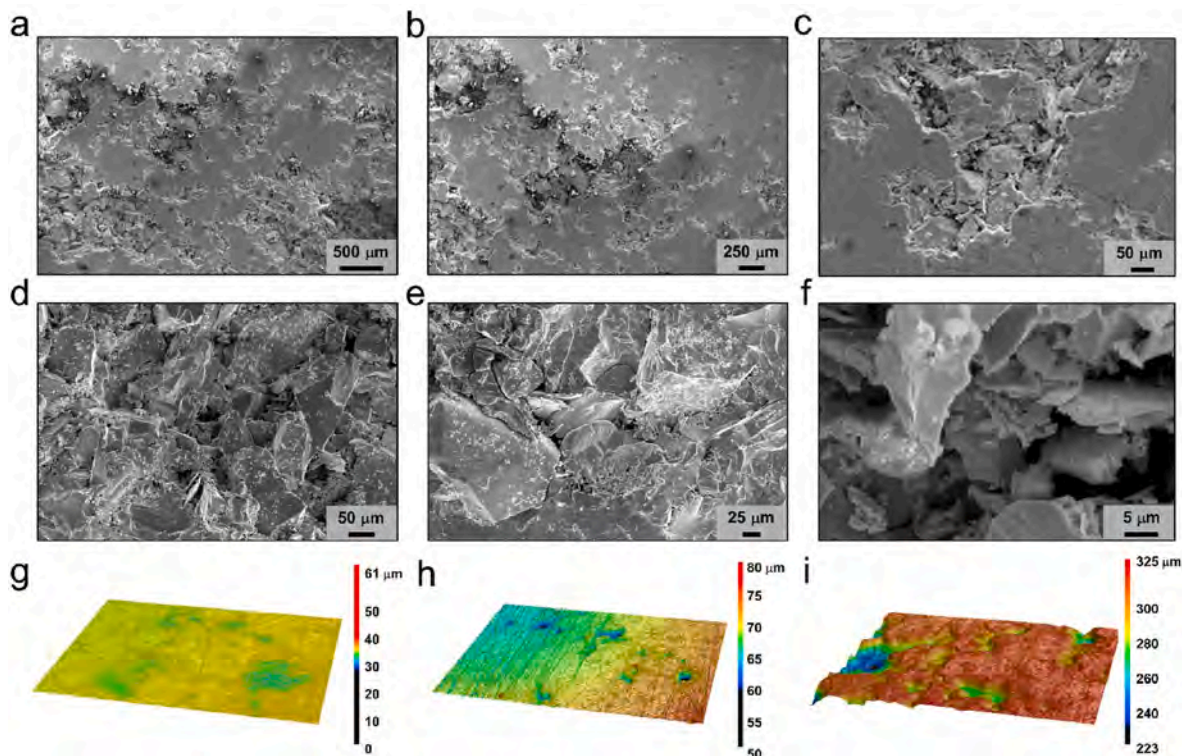

Fig. 5. (a–c) Surface SEM images of bulk composite with 70 vol% Terfenol-D. (d–f) The fracture images of bulk composite with 70 vol% Terfenol-D. (g–h) The Surface CLSM images of Terfenol-D with volume fractions of 10%, 40% and 70%, respectively.

Table 1
Comparison of properties of magnetostrictive composites.

Materials	Processing Time	Processing Pressure	Processing Temperature	Compressive Strength	Magneto-striction	Ref.
Terfenol-D/MG	0.05 h $\leq$	114 MPa	480 K	185 MPa	650 ppm	This Work
Terfenol-D/Polymer	–	500 MPa	423 K	150 MPa	536 ppm	[32]
Terfenol-D/PU	20 h	–	373 K	–	813 ppm	[38]
Terfenol-D/Glass	0.5 h $\geq$	106 MPa	573–873 K	–	640 ppm	[34]
Terfenol-D/Epoxy	13 h $\geq$	8 MPa	343 K	–	900 ppm	[33]
Terfenol-D/Epoxy	2 h	–	353 K	~105 MPa	905 ppm	[39]
Terfenol-D/Epoxy	24 h	9 MPa	353 K	–	720 ppm	[40]
Terfenol-D/Epoxy	20 h	1000 MPa	373 K	–	1005 ppm	[41]
Terfenol-D/Epoxy	1 h	–	343 K	–	1050 ppm	[42]
Terfenol-D/Epoxy	2 h	–	323 K	–	679 ppm	[43]
Terfenol-D/Epoxy	10 h	–	343 K	–	990 ppm	[44]
Terfenol-D/Epoxy	24 h	150 MPa	298 K	–	800 ppm	[45]
Tb-Fe-B/Polymer	–	250 MPa	423 K	100 MPa	493 ppm	[31]
CoO–Fe$_2$O$_3$/Phenol	–	500 MPa	298 K	–	38 ppm	[46]
SmFe$_2$/Fe	–	95 MPa	883 K	–	−443 ppm	[36]
SmFe$_2$/Al	–	95 MPa	883 K	–	−339 ppm	[36]
Fe$_{81}$Ga$_{19}$/Epoxy	1 h	271 MPa	443 K	–	60 ppm	[47]
Fe$_{80}$Ga$_{20}$/Epoxy	24 h	120 MPa	352 K	–	360 ppm	[48]
Sm$_{0.88}$Dy$_{0.12}$Fe$_{1.93}$/Polymer	12 h	–	363 K	–	−186 ppm	[49]
Silicon steel in/Silicon rubber	72 h	–	293 K	–	100 ppm	[50]
Carbonyl iron/Silicon rubber	24 h	–	298 K	–	134 ppm	[51]

the needed time is very long [31–33]. In comparison, the magneto-striction of the MG-bonded composites is comparable with that of polymer-bonded composites, while the compressive strength is better than those of them. Moreover, the fabrication method in this work also only needs a low temperature, while the required time is very short. On the other hand, the SmFe$_2$/metal composites exhibit a magnetostriction of around −450 ppm, and require a very high fabrication temperature up to 883 K. These parameters are both inferior to the results in this work. As a result, this work not only provides a new type of magnetostrictive composites but also show a new method to produce the composites. The new fabrication method by utilizing the MG as the metallic glue is an efficient and convenient way to fabricate bulk magnetostrictive composites with competitive comprehensive properties.

4. Conclusion

In this work, we successfully obtain a new type of MG/Terfenol-D magnetostrictive composites according to a new fabrication method through unitizing the MG to serve as the "metallic glue". The obtained magnetostrictive composites exhibit good comprehensive functional properties, including a large magnetostriction of 650 ppm at 30 kOe and a high compressive strength of 185 MPa. More importantly, the required fabrication condition is very convenient to achieve, which includes a low pressure (114 MPa), a low temperature (480 K), and a short time (3 min). This work provides a new design strategy for the preparation of bulk magnetostrictive materials and may help to develop more MG-based bulk composites with extraordinary functionalities.

Data availability

The data used to support the findings of this study are available from the corresponding author on reasonable request.

Credit authorship contribution statement

Hongji Lin: Methodology, Investigation, Formal analysis, Writing – Original Draft, Visualization. **Jian Yang:** Methodology, Formal analysis. **Hui Zhao:** Investigation. **Junming Gou:** Investigation. **Yiqun Zhang:** Investigation. **Jianan Fu:** Methodology, Investigation, Formal analysis. **Fei Sun:** Methodology, Investigation, Formal analysis. **Wenqing Ruan:** Formal analysis. **Zhenxuan Zhang:** Formal analysis. **Xiong Liang:** Methodology, Formal analysis. **Shuai Ren:** Conceptualization, Writing –

Review & Editing, Funding acquisition, Supervision. **Tianyu Ma:** Resources, Funding acquisition. **Jiang Ma:** Conceptualization, Resources, Writing – Review & Editing, Funding acquisition, Supervision. **Jun Shen:** Resources, Funding acquisition.

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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