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ORIGINAL REPORT



A high-entropy high-temperature shape memory alloy with large and complete superelastic recovery

Shaohui Li^a, Daoyong Cong^a, Zhen Chen^a, Shengwei Li^a, Chao Song^a, Yuxian Cao^a, Zhihua Nie^b and Yandong Wang^a

^aBeijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing, People's Republic of China; ^bSchool of Materials Science and Engineering, Beijing Institute of Technology, Beijing, People's Republic of China

ABSTRACT

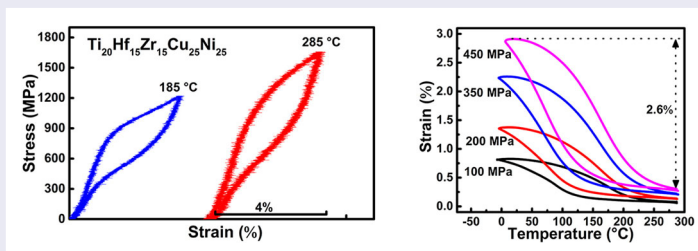
High-temperature shape memory alloys (HTSMAs) show enormous potential for applications as energy conversion devices, actuators and sensors in automotive, aerospace, and energy exploration industries, but they suffer from strength decrease and microstructural instability at elevated temperatures, rendering it difficult to achieve satisfactory high-temperature superelasticity. Here, a novel $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy HTSMA exhibiting large superelasticity with a fully recoverable strain of 4.0% at temperatures up to 285°C is designed with the high-entropy alloy concept by employing the sluggish diffusion and severe lattice distortion effects to suppress thermal softening. This work illuminates the design of novel high-performance functional materials for high-temperature applications.

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KEYWORDS

High entropy alloys; martensitic transformation; superelasticity; shape memory effect; lattice distortion



IMPACT STATEMENT

A novel high-entropy high-temperature shape memory alloy exhibiting large complete superelastic recovery is designed by employing the sluggish diffusion and severe lattice distortion in high-entropy alloys to suppress thermal softening.

1. Introduction

The rapid development of automotive, aerospace, manufacturing, and oil and gas exploration industries urgently demands high-temperature shape memory alloys (HTSMAs) that are used as solid-state energy conversion devices, actuators and sensors at elevated temperatures above 100°C [1,2]. Owing to the reversible phase transformation between the high-temperature, high-symmetry austenitic phase and the low-temperature, low-symmetry martensitic phase, HTSMAs have the potential to recover a large shape change upon removal of applied stress (superelasticity) or upon heating (shape memory effect) [1,3]. Therefore, these alloys exhibit very

promising application prospects in rocket motors, gas turbines, automotive actuators, pressure transducers and so on [1,2].

During the past decades, increasing efforts have been devoted to the exploration of high-performance HTSMAs [1,4–7]. However, there remains a major challenge in developing such alloys with satisfactory high-temperature superelasticity. As is well known, the superelasticity of shape memory alloys occurs only if the yield strength σ_y is higher than the critical stress required for stress-induced martensitic transformation σ_{SIM} ; otherwise, the deformation is dominated by permanent plastic deformation via dislocation slip, leading to irrecoverable

CONTACT Daoyong Cong ✉ dycong@ustb.edu.cn Beijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, People's Republic of China

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strain. Nevertheless, while σ_{SIM} of shape memory alloys increases as the difference between the operating temperature and the transformation temperature increases, σ_y decreases with increasing temperature [1], and thus σ_{SIM} could easily surpass σ_y at high temperatures. Moreover, HTSMAs frequently face the problem of microstructural instability, such as precipitate formation, dislocation recovery and vacancy diffusion at elevated temperatures [8]. The above thermal softening activities render it very difficult to achieve perfect high-temperature superelasticity in HTSMAs. It is imperative to look for viable means to develop high-performance HTSMAs.

The recently proposed high-entropy alloy concept has inspired the exploration of novel alloys with unprecedented properties [9,10]. Particularly, the high-entropy alloy concept can be used to design alloys with excellent resistance to thermal softening [11]. The core effect of sluggish diffusion could effectively suppress the diffusion-controlled processes at elevated temperatures such as vacancy diffusion and grain boundary sliding [12,13] and the severe lattice distortion creates large energy barriers for thermally-activated dislocation motion [13–15], both contributing to the suppression of thermal softening. Meanwhile, the sluggish diffusion does not inhibit the occurrence of diffusionless martensitic transformation [16] and associated functional properties [16–20]. Therefore, it is expected that the high-entropy alloy concept can be used to design HTSMAs with superb superelastic and shape memory properties. Here in this work, we report a novel $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy HTSMA with large perfect superelasticity. This alloy was designed as follows: noticing the conventional $\text{Ni}_{45.3}\text{Ti}_{29.7}\text{Hf}_{20}\text{Cu}_5$ HTSMA reported in the literature [21], we applied the high-entropy alloy concept and obtained the equiatomic composition $\text{Ti}_{25}\text{Hf}_{25}\text{Ni}_{25}\text{Cu}_{25}$, but it shows an austenitic transformation finish temperature A_f of only $\sim 60^\circ\text{C}$; to raise A_f and to further increase the lattice distortion, the composition $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ was finally designed. The as-cast alloy exhibits high-temperature superelasticity with a large fully recoverable strain of 4.0% at temperatures up to 285°C . Furthermore, it shows excellent shape memory effect with a recoverable strain of 2.6%. The present work paves the way for designing advanced HTSMAs with exceptional functionalities at elevated temperatures.

2. Materials and methods

The polycrystalline $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ (at.%) high-entropy alloy was prepared by arc melting in water-cooled copper crucible using high-purity raw materials. The as-cast button ingots were cut into specimens

with different size for different measurements. Differential scanning calorimetry (DSC, NETZSCH 214 Polyma) was used to determine the phase transformation temperatures. The microstructure was examined by using a scanning electron microscope (SEM, Zeiss Supra 55) and a transmission electron microscope (TEM, Tecnai F20) operated at 200 kV. The element distribution was determined by the energy dispersive spectroscopy (EDS) in SEM. The crystal structure at different temperatures was studied by X-ray diffraction (XRD, PW 3040 X'Pert PRO) using $\text{Cu-K}\alpha$ radiation with a wavelength $\lambda = 1.5405 \text{ \AA}$.

The high-temperature superelastic properties were investigated by using a mechanical testing machine (Instron-5966) equipped with a temperature control chamber. Specimens with a size of $\Phi 2 \times 4 \text{ mm}^3$ were cut with the axial direction (also the loading direction) perpendicular to the solidification direction of the button ingot; they show relatively coarse dendritic microstructure and no strong texture parallel to the loading direction is expected. The stress-strain curves were measured in compression mode at different constant temperatures from 185°C to 285°C with a strain rate of $2.1 \times 10^{-4} \text{ s}^{-1}$. The strain was recorded by an advanced non-contacting video extensometer. The shape memory effect was examined by measuring the strain-temperature curves under different constant stresses with a dynamic mechanical analyzer (DMA Q800) equipped with a three-point bending clamp, using a specimen of $25 \times 3 \times 0.5 \text{ mm}^3$.

3. Results and discussion

The backscattered electron images taken in the planes perpendicular to and parallel to the solidification direction of the as-cast $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ button ingot are shown in Figure 1a and b, respectively. Typical dendritic structure can be clearly observed (Figure 1b), which is formed due to the difference in melting points of the constituent elements. Actually, the dendritic structure is commonly observed in the as-cast high-entropy alloys. The element distribution was determined by using EDS (Figure 1c). A quantitative analysis of the compositions of the dendritic and interdendritic regions was also performed (see Table 1 for the results). Clearly, the distribution of the elements is not homogeneous across the dendritic and interdendritic regions (Figure 1c). The dendritic region with bright contrast in the backscattered electron image is rich in Hf and Ni and lean in Ti, Zr and Cu, while the interdendritic region with dark contrast is rich in Ti, Zr and Cu and lean in Hf and Ni (Table 1). The dendritic microstructure may increase the strength and decrease the ductility.

The XRD patterns collected at 200°C and -100°C are shown in Figure 2a. At 200°C , the major phase is the cubic

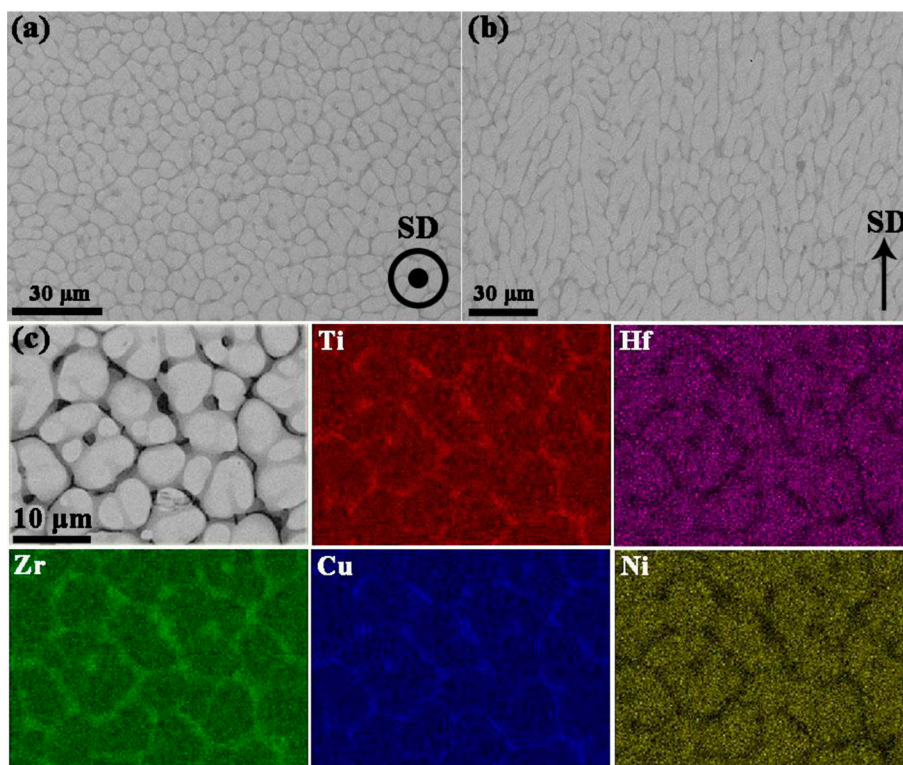


Figure 1. Backscattered electron images taken in the planes (a) perpendicular to and (b) parallel to the solidification direction (SD) of the button ingot. (c) EDS maps of the elements Ti, Hf, Zr, Cu and Ni, taken from the area the backscattered electron image of which is shown in the upper left panel.

Table 1. Average chemical compositions (at.%) of the dendritic and interdendritic regions for the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ alloy.

Region	Ti	Hf	Zr	Cu	Ni
Dendritic region	17.81	17.15	13.67	21.76	29.61
Interdendritic region	21.28	11.71	16.47	28.90	21.64

B2 austenite, with a small amount of coexisting monoclinic B19' martensite. The lattice constant of the cubic B2 austenite is $a_A = 3.172 \text{ \AA}$, and the lattice parameters of the monoclinic B19' martensite are $a_M = 3.331 \text{ \AA}$, $b_M = 4.216 \text{ \AA}$, $c_M = 4.781 \text{ \AA}$, and $\beta = 100.3^\circ$. When the temperature is decreased to -100°C , although there still exists residual austenite, the intensities of the martensitic peaks become stronger and several new martensitic peaks appear (Figure 2a), implying that the volume fraction of martensite increases during cooling. Therefore, the present high-entropy alloy shows martensitic transformation during cooling.

The DSC curve for the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy is depicted in Figure 2b. Exothermic and endothermic peaks corresponding respectively to the martensitic and reverse austenitic transformations can be clearly observed, demonstrating the reversibility of the transformation. The martensitic transformation start temperature M_s and the A_f are 101 and 179°C ,

respectively. The present high-entropy alloy shows a relatively large thermal hysteresis ($A_f - M_s = 78^\circ\text{C}$) and broad phase transformation intervals, which, on the one hand, could be due to the inhomogeneous element distribution (Figure 1 and Table 1) and, on the other hand, could be ascribed to the imperfect geometric compatibility between austenite and martensite, as presented below. As mentioned above, the interdendritic region is rich in Ti, Zr and Cu and lean in Hf and Ni; it is possible that the interdendritic region is non-transformable during cooling since our experience obtained during composition tuning suggests that both substitution of Cu for Ni and substitution of Ti or Zr for Hf lead to significant decrease of transformation temperature. According to the geometric nonlinear theory of martensite, the thermal hysteresis is to a large extent governed by the geometric compatibility at the interface between martensite and austenite [22–24]. The geometric compatibility can be characterized by the middle eigenvalue λ_2 of the transformation stretch tensor $\mathbf{U}$. If λ_2 is close to 1, the stress transition layer at the interface between martensite and austenite could be greatly reduced or even eliminated, and thus the martensitic transformation has a very narrow thermal hysteresis [23]. Based on the lattice parameters of martensite and austenite determined from the XRD experiments (Figure 2a), the

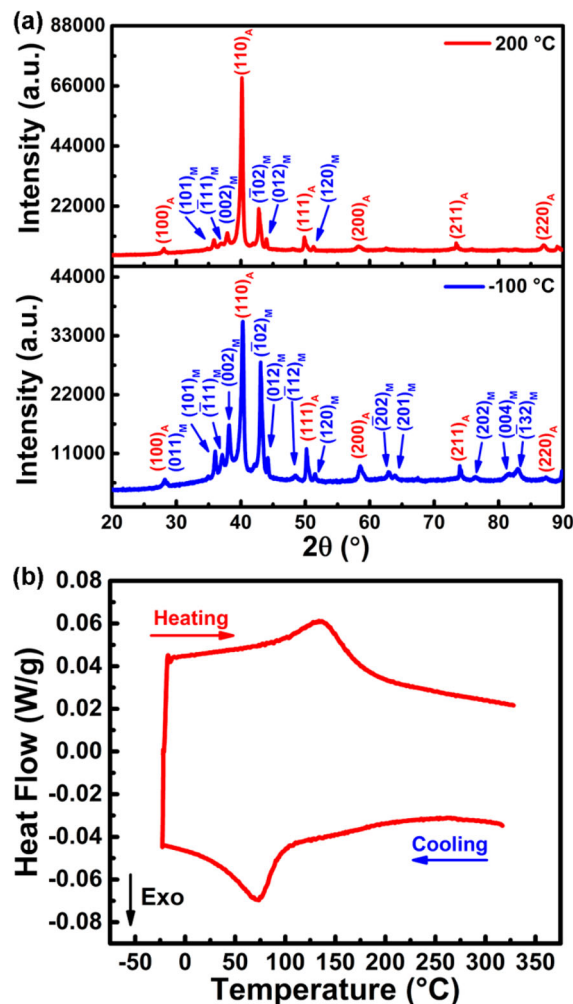


Figure 2. (a) X-ray diffraction patterns measured at 200°C and –100°C. The letters ‘A’ and ‘M’ in the indices denote austenite and martensite, respectively. (b) DSC curve recorded with a heating and cooling rate of 10°C/min.

λ_2 is calculated to be 0.9584. This value deviates from 1, which indicates that the geometric compatibility is not very good in the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy. In spite of the relatively large thermal hysteresis ($A_f - M_s = 78^\circ\text{C}$), the martensitic transformation in the present $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy is still thermoelastic in nature since the thermal hysteresis is below 100°C [1,3]. This serves as the basis for the functional properties demonstrated below.

To examine the high-temperature superelasticity, we measured the stress-strain curves at different constant temperatures ranging from 185°C to 285 °C (above A_f). Strikingly, the present $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy exhibits perfect high-temperature superelasticity with large recovery. The applied strain of 4% can be fully recovered after unloading and thus the recoverable strain is as large as 4.0%. The maximum recoverable strain may be even larger if a higher strain is applied. The

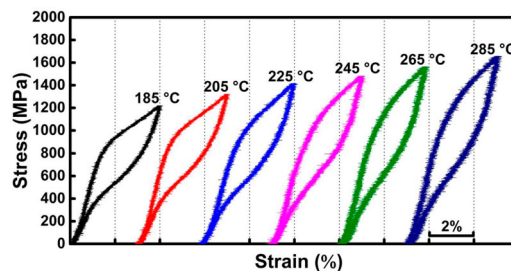


Figure 3. Stress-strain curves recorded at different constant temperatures ranging from 185°C to 285°C.

maximum temperature at which the large perfect superelasticity occurs is as high as 285°C. This remarkable perfect high-temperature superelasticity has never been achieved before in high-entropy alloys. It is worth mentioning that the precipitation strengthened conventional $\text{Ni}_{50.3}\text{Ti}_{29.7}\text{Hf}_{20}$ HTSMA shows a recoverable strain of 3.0% in the temperature range 180–220°C [25]. As displayed in Figure 3, the superelastic stress-strain response of the present high-entropy alloy is dependent on the test temperature; σ_{SIM} increases with the increase of temperature. Noteworthy, the applied stress is as high as 1.65 GPa at 285°C and the present alloy still exhibits perfect high-temperature superelasticity, suggesting that the yield strength is higher than 1.65 GPa at this temperature. This confirms that the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy shows high yield strength at high temperatures.

TEM observation on the fine microstructure of the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy HTSMA was conducted at room temperature. Figure 4a shows a bright-field TEM image and Figure 4b displays the selected area electron diffraction (SAED) pattern taken from the area enclosed by the circle in Figure 4a. No nanoscale precipitates can be observed from Figure 4a and there are no diffractions from precipitates in the SAED pattern. In fact, the SAED pattern contains diffractions from both B19' martensite and B2 austenite, consistent with the XRD results (Figure 2a). In addition, the (011) type I twinning of B19' martensitic variants can be clearly identified (Figure 4b). As indicated from the high-resolution TEM image in Figure 4c and the inverse fast Fourier transform image in Figure 4d, the atomic positions deviate from the ideal lattice points and a large amount of kinks and bends in the lattice layers are visible (Figure 4d), similar to those observed in a $\text{Nb}_{25}\text{Mo}_{25}\text{Ta}_{25}\text{W}_{25}$ high-entropy alloy [26]. This clearly demonstrates the severe lattice distortion in the present $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ alloy. The severe lattice distortion is caused by the difference in atomic radii of constituent elements (namely the atomic size misfit); particularly, in the present high-entropy alloy the atomic sizes of Zr

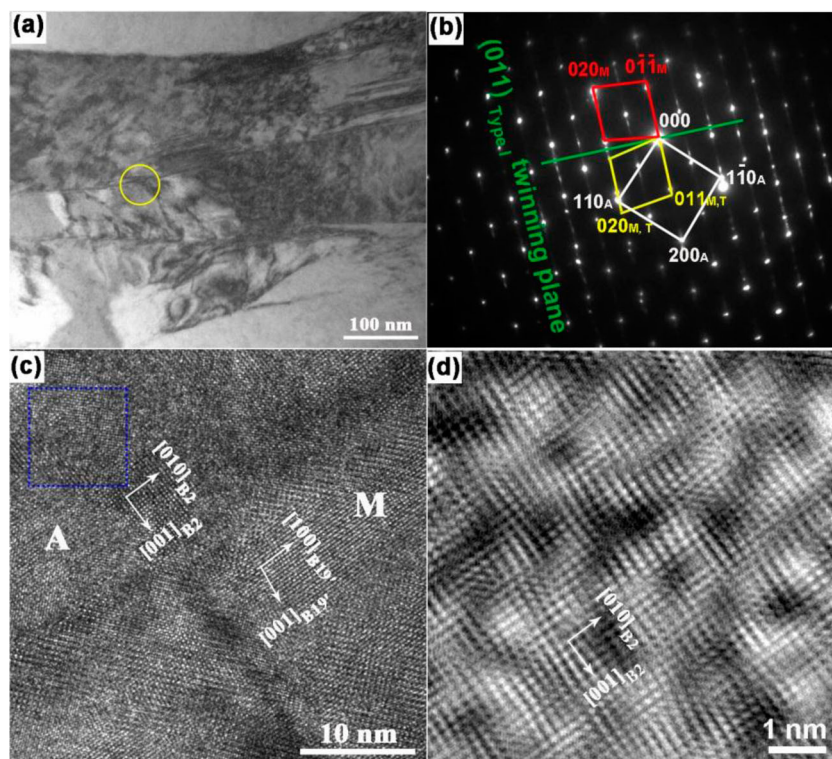


Figure 4. (a) Bright-field TEM image. (b) Selected area electron diffraction pattern taken from the area enclosed by the circle in (a), which is from the dendritic region. The subscripts ‘A’, ‘M’ and ‘T’ in the indices denote austenite, martensite and twin, respectively. (c) High-resolution TEM image taken from an area with coexisting austenite (A) and martensite (M). The A and M regions were identified from the corresponding fast Fourier transform patterns (not shown) of these regions. (d) Inverse fast Fourier transform image for the area enclosed by the rectangle in (c).

and Hf are significantly larger than Ti, Ni and Cu. The severe lattice distortion results in remarkable solid solution hardening and it also produces large energy barriers for (and thus reduces) thermally-activated dislocation motion. As a result, the present $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy shows high yield strength at high temperatures and thus large perfect high-temperature superelasticity. Moreover, it is possible that the sluggish diffusion in the high-entropy alloy suppresses the diffusion-controlled processes including vacancy diffusion and grain boundary sliding [12,13], and this may also contribute to the large perfect high-temperature superelasticity.

Since shape memory devices usually operate with a restoring bias force during service, the shape memory effect was investigated by load-biased thermal cycling tests. Figure 5a shows the strain-temperature curves recorded with the three-point bending mode during load-biased thermal cycling under different constant stresses for the $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy. The determination of the recoverable strain ε_{rec} and irrecoverable strain ε_{irr} is illustrated in this figure. The ε_{rec} and ε_{irr} are plotted as a function of stress in Figure 5b. The present alloy exhibits perfect shape

memory effect with no ε_{irr} when the stress is in the range of 100–450 MPa (Figure 5a). The ε_{rec} is 0.7% under 100 MPa and it increases to 2.6% under 450 MPa. The increase in ε_{rec} with increasing applied stress is due to the increase in volume fraction of preferably selected martensitic variants. Under the higher stress level of 550 MPa, a small ε_{irr} (0.15%) appears and the ε_{rec} decreases to 2.4% (Figure 5b). This could be attributed to the occurrence of plastic deformation by dislocation slip and martensite stabilization under this stress level. Noteworthy, the present $\text{Ti}_{20}\text{Hf}_{15}\text{Zr}_{15}\text{Cu}_{25}\text{Ni}_{25}$ high-entropy alloy exhibits a maximum ε_{rec} of as high as 2.6%, which is actually a high shape memory strain. Figure 5c shows the stress dependence of transformation temperatures M_s and A_f extracted from the strain-temperature curves (Figure 5a). It is clear that the phase transformation temperatures increase almost linearly with the stress and the M_s and A_f increase from 109°C to 153°C and from 196°C to 231°C, respectively, as the stress increases from 100 MPa to 550 MPa. Actually, the relationship between the applied stress and the transformation temperatures T follows the Clausius-Clapeyron relation: $d\sigma/dT = -\Delta H/T_0\varepsilon_{\text{tr}}$, where ΔH is the transformation enthalpy change, T_0 is the equilibrium temperature,

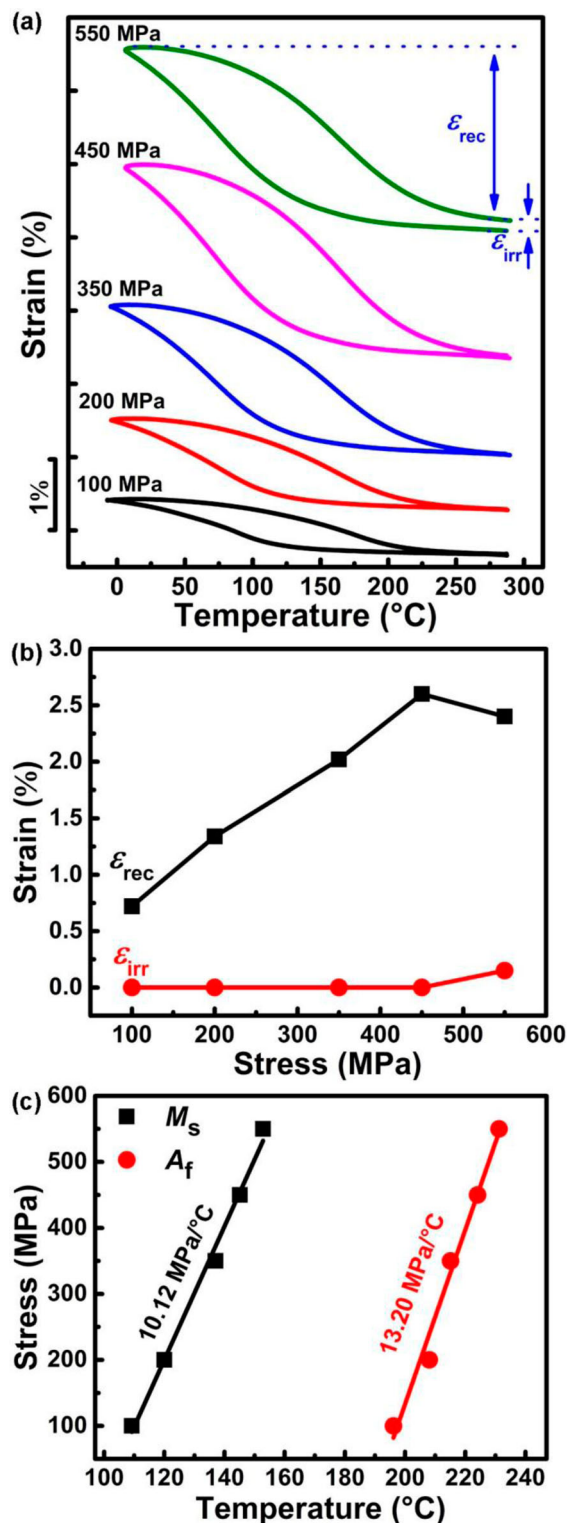


Figure 5. (a) Strain-temperature curves measured with the three-point bending mode during load-biased thermal cycling under different stresses. The determination of the recoverable strain ϵ_{rec} and irrecoverable strain ϵ_{irr} is illustrated. (b) ϵ_{rec} and ϵ_{irr} plotted as a function of stress. (c) Stress dependence of M_s and A_f .

and ϵ_{tr} is the transformation strain. Based on the linear fittings of the experimental data extracted from Figure 5a, the $d\sigma/dT$ values for M_s and A_f for the present

Ti₂₀Hf₁₅Zr₁₅Cu₂₅Ni₂₅ high-entropy alloy are 10.12 and 13.20 MPa/°C, respectively, which are comparable with those reported for traditional Ni-Ti-Pd (5.9–8.4 MPa/°C) [27] and Ni-Ti-Hf (8–12.5 MPa/°C) [28] HTSMAs.

4. Conclusions

A novel Ti₂₀Hf₁₅Zr₁₅Cu₂₅Ni₂₅ high-entropy high-temperature shape memory alloy with large perfect superelasticity has been designed by employing the sluggish diffusion and severe lattice distortion effects in high-entropy alloys. The as-cast alloy exhibits high-temperature superelasticity with a fully recoverable strain of as high as 4.0% at temperatures up to 285°C. Furthermore, it shows excellent shape memory effect with a recoverable strain of 2.6% under a stress of 450 MPa. This study provides a strategy for developing high-performance high-temperature functional materials for applications at elevated temperatures in automotive, aerospace, manufacturing, and energy exploration industries.

Disclosure statement

No potential conflict of interest was reported by the authors.

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ORCID

Shaohui Li  <http://orcid.org/0000-0003-3078-0338>

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