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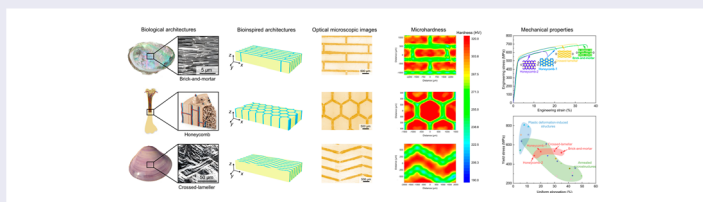
Bioinspired strategy to break strength-plasticity trade-off in high-entropy alloy

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ABSTRACT

Constructing bioinspired architectures in metallic materials is an effective way to overcome the inherent strength and plasticity trade-off dilemma. However, conventional processing approaches are difficult to finely tailor the microstructures and mechanical properties of bioinspired architectures. Here, using laser remelting technology, we finely and controllably fabricate three sophisticated bioinspired architectures in a high-entropy alloy (HEA). The bioinspired HEA with brick-and-mortar architecture shows an excellent yield strength of ~ 520 MPa with a large uniform elongation of $\sim 35\%$. Such enhanced strength-plasticity synergy is mainly attributed to the hetero-deformation between the hard and soft regions of bioinspired architectures.



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KEYWORDS

Bioinspired architecture; high-entropy alloy; laser remelting technology

1. Introduction

Metallic materials play crucial roles in various industrial and advanced applications. Among the key mechanical properties of metallic materials, strength and plasticity stand out as crucial factors at room temperature. However, there exists a well-known trade-off dilemma between strength and plasticity [1–9]. Many traditional strategies to strengthen metallic materials, such as precipitate strengthening and dislocations strengthening, always come at the expense of plasticity [10–13]. An urgent challenge, therefore, is how to adopt brand-new alternative strategies to simultaneously enhance the strength and ductility of metallic materials.

To overcome this trade-off problem, nature is undoubtedly our best mentor. Organisms have evolved over billions of years to utilize a small number of individual constituents with comparatively meager mechanical properties, fine-tuning their architectures to result in exceptional mechanical properties [14–16]. The constituents are intricately arranged at various scales, from molecular to macro, resulting in complex architectures with

diverse internal gradients and interfaces [17]. Drawing insights from natural organisms, the bioinspired design of architectures has emerged as a promising strategy for enhancing the performances of man-made metallic materials [18]. The bioinspired design strategy offers opportunities for synergistically improving both strength and plasticity. While bioinspired designs have been successfully implemented in polymers and polymer-based composites due to their ease of processing, challenges remain in extending these principles to metallic materials [19–21]. Specifically, constructing bioinspired architectures of metallic materials is particularly challenging due to the complexities of controlling microstructures and mechanical properties during conventional manufacturing processes.

Recently, several bioinspired designs, including bimodal, harmonic, lamellar, and gradient, have been realized in metallic materials. These bioinspired metallic materials are normally prepared by complicated thermomechanical routes involving severe plastic deformation [22, 23] or dynamic plastic deformation [24, 25], followed by

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an annealing treatment. To obtain excellent mechanical properties during bioinspired metallic materials processing, one needs to regulate and optimize the volume fraction, morphology, and spatial variation of the bioinspired designs, as these variables might strongly influence the mechanical properties. Nevertheless, traditional methods make it difficult to control the above factors accurately, particularly at elevated temperatures and pressures. It is necessary to invent novel processing methods to prepare sophisticated bioinspired metallic materials.

Laser remelting technology is promising for the construction of sophisticated bioinspired architectures due to its unique advantages of high machining accuracy, high laser flexibility, small heat affected region, etc. [26–29]. Here, we harness high-entropy alloys (HEAs) [30, 31] as the model materials and use laser remelting technology to produce a series of sophisticated bioinspired architectures. Three types of bioinspired architectures were well-designed: (i) the brick-and-mortar architecture simulating nacre [32–34], (ii) the honeycomb architecture simulating bone [35–37], and (iii) the crossed-lamellar architecture simulating *saxidomus purpuratus* shells [38]. All these architectures, as illustrated in Figure 1, have been shown to exhibit remarkable mechanical properties in their corresponding biological materials. We assess and compare the mechanical properties of these composites, specifically focusing on strength and plasticity. The fabricated bioinspired HEA with brick-and-mortar architecture exhibits a favorable yield strength of ~ 520 MPa with an excellent uniform elongation of $\sim 35\%$. The synergetic strengthening is mainly attributed to the hetero-deformation associated with the sophisticated bioinspired heterogeneous architecture. The microcracks are strictly arrested at the interface of the soft and hard regions, consuming significant mechanical energy and effectively prolonging the plasticity.

2. Materials and methods

Bulk HEA ingots with a nominal composition of $\text{Al}_{0.5}\text{Cr}_{0.9}\text{FeNi}_{2.5}\text{V}_{0.2}$ were prepared by vacuum induction electromagnetic levitation melting a mixture of pure metals (purity, > 99.9 wt.%) in a high-purity argon atmosphere. The ingots were remelted at least eight times to ensure compositional homogeneity. To reduce micro-segregations from the casting process, the samples were homogenized at 1150°C for 24 h and then water-quenched to room-temperature. Subsequently, the samples were aged at 700°C for 1 h followed by furnace cooling. Plates with dimensions of $100 \times 20 \times 1$ mm³ were cut from the samples for laser remelting experiments. A 1064-nm continuous-wave

fiber laser (FF-M180 machine, Fastform) was used to implement the remelting experiment. The spot size was about $125\text{ }\mu\text{m}$. The laser power and scanning speed were 400 W and 75 mm/s, respectively.

Optical microscopic (OM) images of bioinspired architecture samples were taken using an inverted microscope with a 4XC20-V camera. Scanning electron microscope (SEM) and electron backscatter diffraction (EBSD) measurements were performed with a Zeiss Supra55/Oxford EBSD system. The EBSD data were interpreted using Channel 5.0. Transmission electron microscopy (TEM) measurements were performed on a JEOL-2100F high-resolution transmission electron microscope. Vickers microhardness tests were performed using an HVD-1000IS microhardness tester with a load of 300 g and a dwell time of 15 s. Quasi-static uniaxial tensile tests were performed on an MTS C45.105EY testing machine at a strain rate of $2 \times 10^{-3} \text{ s}^{-1}$. The tests were repeated at least three times for each type of sample.

3. Results and discussions

To explore the variance of non-remelted and remelted regions, we conducted a multiple-scale observation and analysis of the microstructure (Figure 2), where the brick-and-mortar architecture is taken as an illustration (these characteristics were the same as the three architectures). A low-magnification EBSD inverse pole figure (IPF) map of the sample with brick-and-mortar architecture is shown in Figure 2(a). The remelted regions reveal an unchanged grain size compared with the non-remelted grains. Figures 2(b-1) and (b-2) display the typical EBSD IPF and phase maps of the non-remelted region. The microstructure of this region is composed of a primary FCC phase and a minor BCC phase, with the minor BCC phase distributed at the grain boundaries. Bright-field TEM (BF-TEM) image of the non-remelted region is shown in Figure 2(b-3), marking the primary FCC phase and minor BCC phase. The selected-area electron diffraction (SAED) pattern of the $[011]$ zone axis corresponding to the FCC phase is presented in the upper right inset, exhibiting reflections of an ordered FCC (L_{12} structure) and suggesting that the region consists of the FCC matrix and high-content L_{12} strengthening phase. Subsequently, we analyzed the microstructure of remelted region. The IPF and phase maps reveal the presence of only the FCC phase (Figures 2(c-1) and (c-2)), indicating that the accelerated cooling rate during remelting inhibits the precipitation of BCC phase. We used TEM characterization to provide direct evidence to further verify the precipitates at the grain boundaries of the remelted region. BF-TEM image of the grain boundary indicates

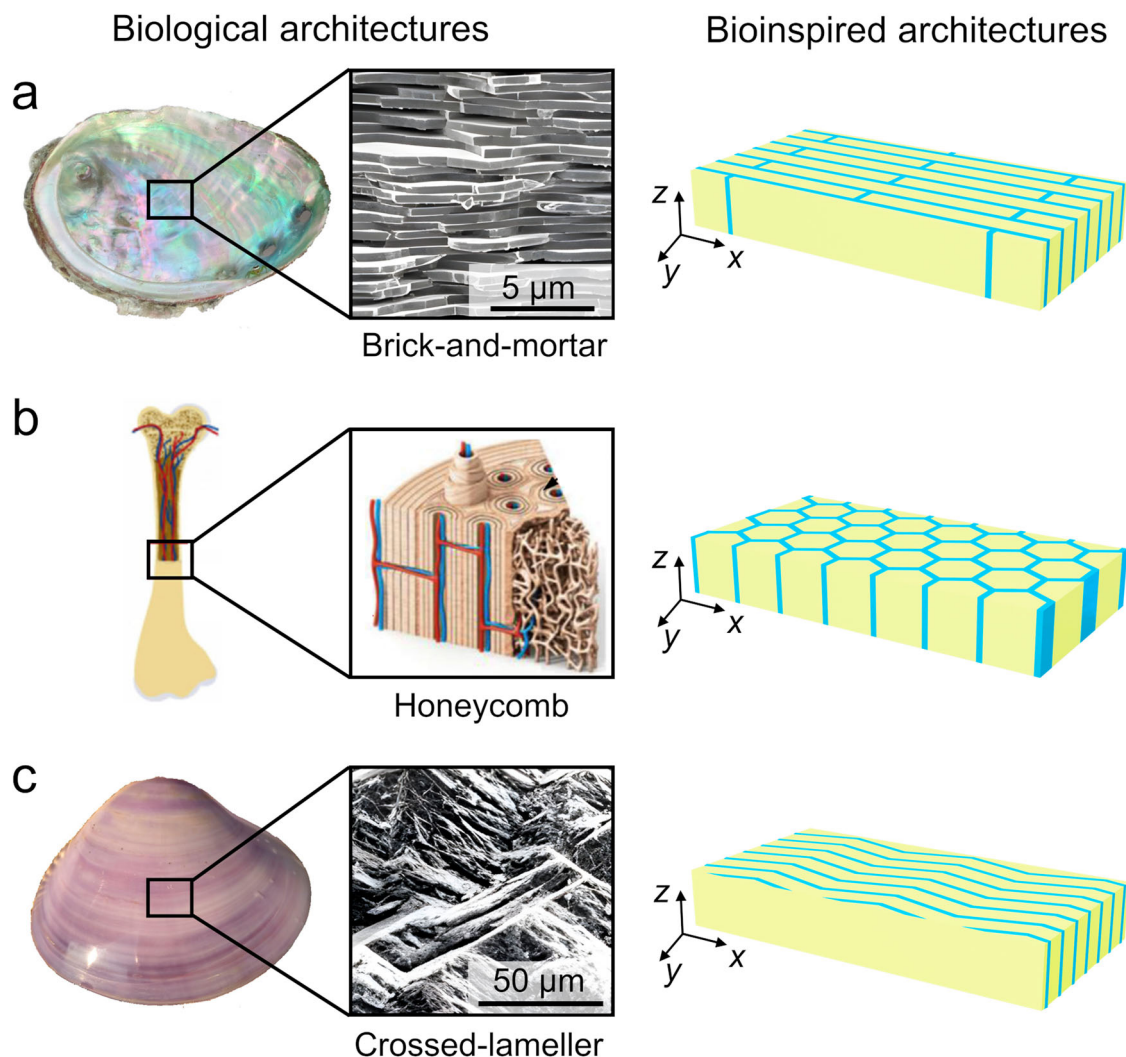


Figure 1. Design of bioinspired architectures. The structures of representative biological prototypes, respectively the shell of abalone[34], the bone osteon of compact bone[19], and the shell of *saxidomus purpuratus*[38] for the (a) brick-and-mortar, (b) honeycomb, and (c) crossed-lamellar architectures, are also presented for comparison. Schematic bioinspired architectures models of biological prototypes are shown on the right. The blue and yellow colors in the schematics represent the soft and hard phases, respectively.

that no precipitates are observed (Figure 2(c-3)). Additionally, the SAED shows in the inset of Figure 2(c-3) demonstrates that no reflections of an ordered FCC ($L1_2$ structure).

Figure 3 shows the OM images of cross-sections with brick-and-mortar (Figure 3(a)), honeycomb (Figure 3(c)), and crossed-lamellar architectures (Figure 3(e)). Clearly, the key features of the characteristic dimensions and spatial arrangements in these biological materials are retained and well reproduced into the bioinspired HEAs. The bioinspired HEAs have two different contrast regions, wherein the dark yellow remelted regions are embedded in a light yellow non-remelted regions matrix.

The mechanical properties of alloys can be predicted using Vickers hardness, which is defined as the Tabor relationship. To demonstrate that laser remelting

technology can effectively control the mechanical properties, we carried out microhardness experiments on three types of bioinspired architectures. Figure 3 shows Vickers hardness mappings of three bioinspired architectures at room-temperature. The Vickers hardness mappings clearly observe that a strong microhardness variance exists in the bioinspired HEAs, which are 308 and 205 HV in the non-remelted and remelted regions, respectively. Notably, a change in the laser scanning path does not affect the microhardness in the laser remelted regions. The significant difference in microhardness is owing to the difference in phase composition between non-remelted and remelted regions. In non-remelted regions, high hardness results from the precipitation strengthening effect of BCC and $L1_2$ phases, whereas no precipitates are observed in remelted regions, leading to low hardness.

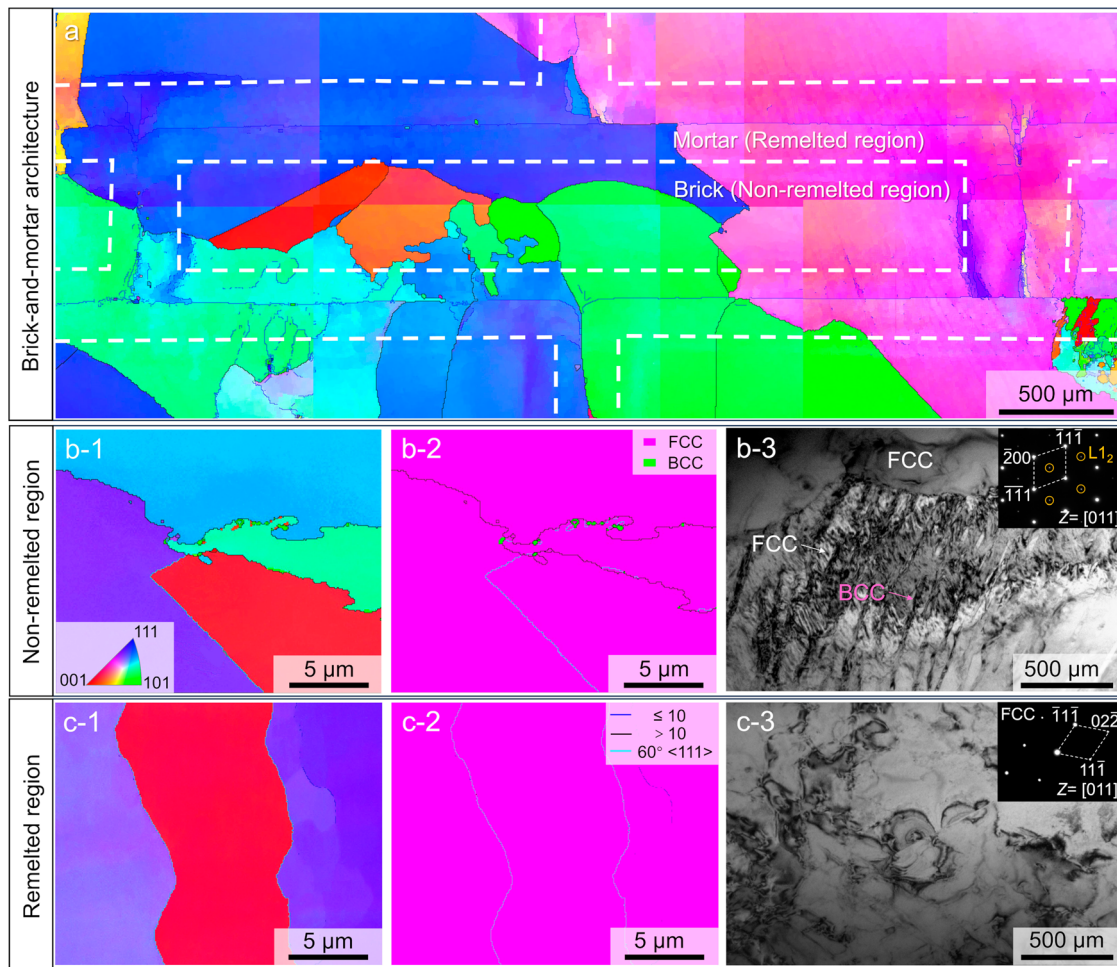


Figure 2. Microstructural characteristics of bioinspired HEAs. (a) EBSD IPF map of bioinspired HEAs with Brick-and-mortar architecture. (b-1) — (b-3) Microstructures of non-remelted region. (b-1) EBSD IPF map of non-remelted region. (b-2) The corresponding phase map showing the FCC phase in pink and BCC phase in green. In (b-2), the black lines represent high-angle grain boundaries ($> 10^\circ$), the blue lines represent small-angle grain boundaries ($\leq 10^\circ$), and the cyan lines represent $60^\circ < 111 \rangle$ twin boundary. (b-3) BF-TEM micrograph of the non-remelted region. The inset showing SAED of the FCC matrix. (c-1) — (c-3) Microstructures of remelted region. (c-1) EBSD IPF map of remelted region. (c-2) The corresponding phase map showing only the FCC phase in remelted region. (c-3) BF-TEM micrograph of the remelted region.

Figure 4(a) shows the engineering stress–strain curves of tensile tests for three types of bioinspired samples. The three types of bioinspired architectures possess approximately the same yield strength in tension, but the plasticity differs significantly. Notably, bioinspired HEA with brick-and-mortar architecture exhibits a remarkable combination of high yield strength (520 MPa) and large uniform elongation ($\sim 35\%$). To prove the superiority of bioinspired architectures, we compared the present bioinspired HEAs with other reported homogeneous HEAs with similar alloy compositions [39–44]. As shown in Figure 4(b), the present bioinspired HEAs with brick-and-mortar architecture visibly stands out in the general trend of strength-ductility trade-off for homogeneous alloys, indicating a superior strength-ductility synergic action.

The above experimental results show that the bioinspired HEA with brick-and-mortar architecture has an outstanding combination of strength and plasticity. In order to explore the mechanical behaviors of this bioinspired HEA in detail, its microstructural evolution was interrupted at some certain strain levels during the ambient deformation process and observed by SEM and EBSD, as presented in Figures 5 and S1.

Prior to deformation (Figure S1(a)), the macroscopic SEM images of the bioinspired HEA can clearly observe the non-remelted and remelted regions of the brick-and-mortar architecture. In addition, a small number of pre-existing dislocations are perceived in remelted regions (Figures 5(a) and (c)). The pre-existing dislocations are derived from thermal stresses developed during the laser remelting process. At a strain of 7% (Figure S1(b)), a few

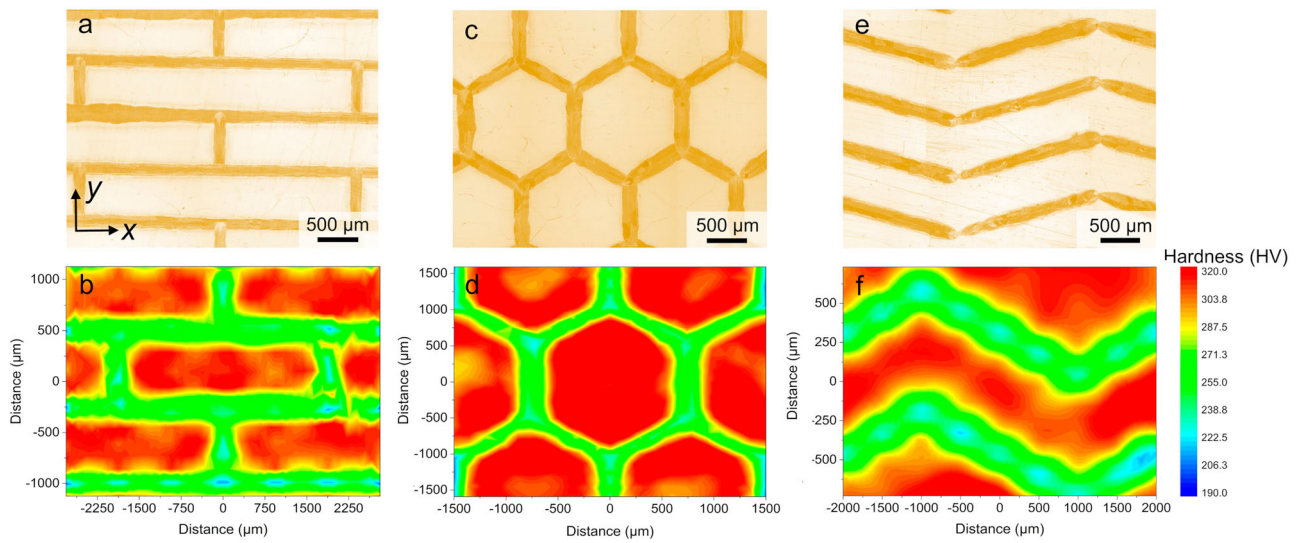


Figure 3. Optical microscopic images and Vickers hardness mappings of three bioinspired HEAs. (a) and (b) Brick-and-mortar architecture. (c) and (d) Honeycomb architecture. (e) and (f) Crossed-lamellar architecture.

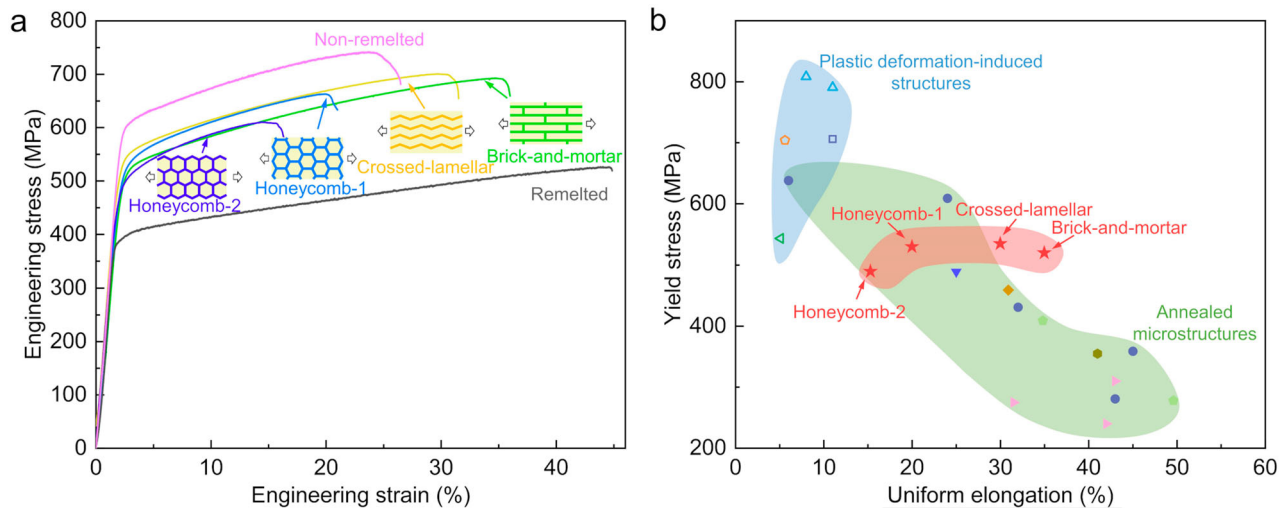


Figure 4. Mechanical properties of HEAs. (a) Uniaxial tensile engineering stress-strain curves of the HEAs with different architectures. (b) A comparison of yield stress and uniform elongation of present bioinspired HEAs with other reported homogeneous HEAs with similar alloy systems [39–44].

slip bands are observed in remelted and near remelted regions, suggesting that the remelted regions preferentially undergo yielding. By contrast, no obvious dislocation is detectable in non-remelted regions, as reflected by the KAM map in Figure 5(d), which indicates that these regions still deform elastically. Furthermore, the KAM map indicates that the dislocation density increases gradually with the increment of strain, especially in the remelted region. The above results demonstrates that hetero-deformation occurs between the remelted and non-remelted regions. As the strain increases to 14% (Figure S1(c)), many slip bands could be observed in the remelted and non-remelted regions, revealing that both the two regions plastically deform. At a strain of 21%

(Figure S1(d)), the microcracks are observed to nucleate from the non-remelted regions. With further increasing the strain to 28% (Figure S1(e)), the microcracks strictly arrested at the interfaces of the remelted and non-remelted regions instead of expanding to the formation of large cracks. After the sample fracture (Figure S1(f)), the fracture surface of the bioinspired HEA with brick-and-mortar architecture clearly exhibits the crack propagating along a tortuous path.

The above results demonstrate that the distinctive behavior of the bioinspired HEA with brick-and-mortar architecture stems from its sophisticated architecture. To better understand the underlying origin of the remarkable combination of strength and plasticity in bioinspired

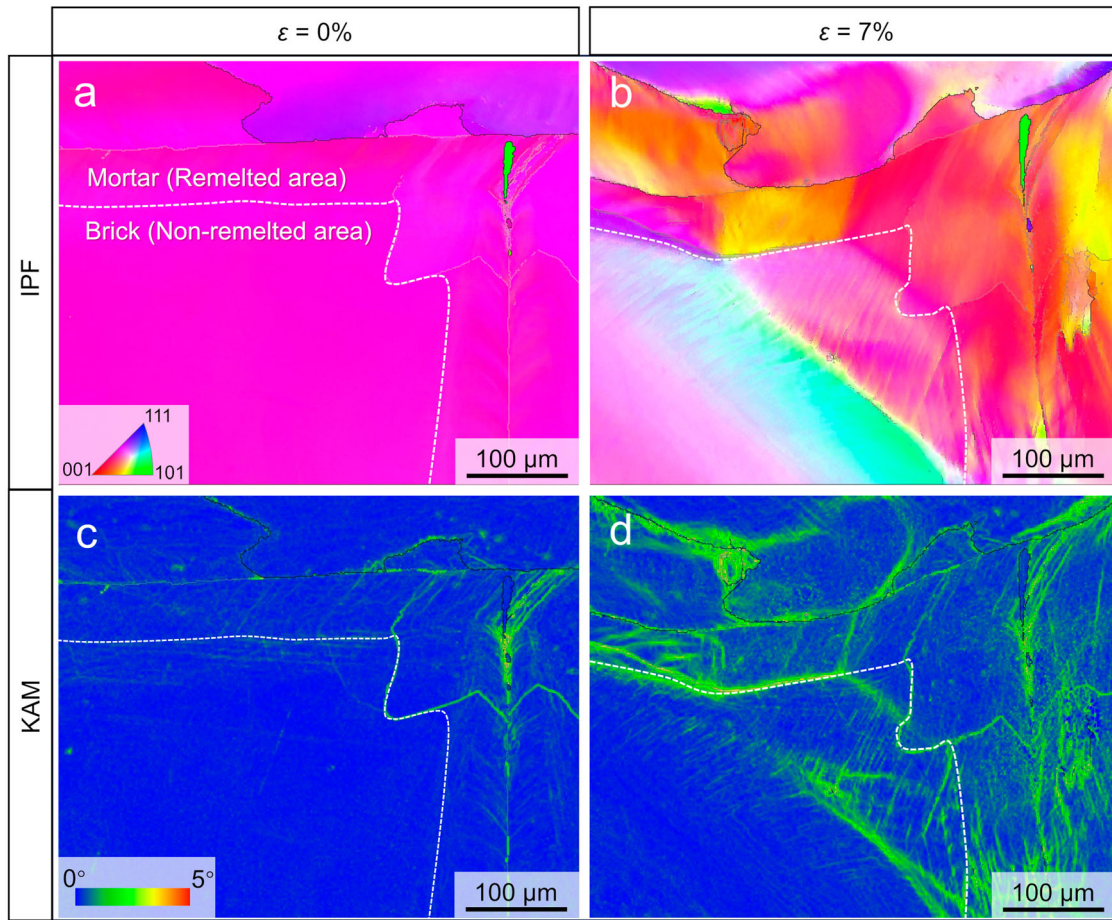


Figure 5. Microscopic deformation structures of bioinspired HEA with brick-and-mortar architecture. (a) and (b) EBSD IPF maps of brick-and-mortar architecture at tensile strains of 0% (a) and 7% (b). (c) and (d) KAM maps of brick-and-mortar architecture at tensile strains of 0% (c) and 7% (d).

HEA with brick-and-mortar architecture, the strengthening and toughening mechanisms of this architecture will be further explored.

As shown in Figure 4, bioinspired HEA with brick-and-mortar architecture exhibits an excellent yield strength of ~ 520 MPa. To illustrate the underlying reasons for the high yield strength, the yield strength of bioinspired HEA is firstly evaluated by applying the rule of mixtures (ROM)[32]: $\sigma_{y,ROM} = v_{soft} \times \sigma_{soft} + v_{hard} \times \sigma_{hard}$, where $v_{hard} = 54\%$ and $v_{soft} = 46\%$ are the volume fractions of non-remelted regions and remelted regions, respectively. $\sigma_{hard} = 605$ MPa and $\sigma_{soft} = 382$ MPa are the yield strength of the non-remelted and the entire remelted samples, respectively. Then the yield strength of the bioinspired HEA with brick-and-mortar architecture is evaluated to be approximately $\sigma_{y,ROM} = 502$ MPa, lower than the actual measured value (~ 520 MPa). The synergetic strengthening, i.e. the extra strengthening beyond the ROM prediction, is then predicted to originate from the mutual restraint effect between the non-remelted and remelted regions.

These non-remelted and remelted regions endure different levels of plastic strain during deformation due to inconsistent strength (Figure 3(b)). As illustrated in Figure S2(a), under relatively low stress, both the hard non-remelted and soft remelted regions underwent elastic deformation. Upon further loading, as the entire sample reaches macroscopic yielding, the soft remelted regions yield first, while the hard non-remelted regions continue to experience elastic deformation (Figure S2(b)). The deformation of the soft remelted regions is confined by the hard non-remelted regions, leading to a strain gradient near their interfaces. This strain gradient induces abundant geometrically necessary dislocations (GNDs) to be activated and piled up on their interfaces, as observed by the kernel average misorientation (KAM) results shown in Figure 5(d). The build-up of GNDs at the interface hinders dislocation slipping within the soft remelted regions, providing internal long-range back stress. The contribution of internal long-range back stress to the synergetic strengthening can be confirmed by the loading-unloading-reloading tensile test,

as presented in Figure S3. The curve with prominent hysteresis loops testifies to a notable Bauschinger effect [45], with the back stresses increasing from ~ 460 MPa to ~ 596 MPa with loading [46]. Clearly, such strong back-stress hardening reflects the beneficial effect of brick-and-mortar architecture by laser remelting technology.

On the basis of these descriptives, we now discuss the structural origins responsible for the large plasticity of bioinspired HEA with brick-and-mortar architecture. The large plasticity of bioinspired HEA comes largely from the following two aspects: First, the hetero-deformation in this architecture results in high work hardening and the consequent large plasticity. As described above, the remelted regions are softer than the non-remelted regions, leading to the remelted regions yield first and deform plastically during the deformation process (Figure 5(d)). As stress increased, the hard non-remelted regions undergo progressive yielding (Figure S1(c)). This hetero-deformation of the intricate bioinspired architecture induces abundant GNDs to be activated and piled up on the interfaces of hard non-remelted and soft remelted regions [47]. These GNDs accommodation due to strain gradient near interfaces will contribute to the additional work hardening [48], which is the key factor in obtaining high plasticity.

Second, microcracks in the hard regions are strictly arrested at the interface of the soft and hard regions. As a result, the fracture of the bioinspired architecture can be retarded. Due to the intricate bioinspired architecture simulating nacre, the microcracks are limited to the local hard regions and do not extend into the soft or the neighboring hard regions [49, 50]. Additionally, at the microcrack tip, the soft regions deconcentrate stress over the hard regions (Figure S1(e)). These controlled microcracks contribute to reducing stress concentration, consuming significant mechanical energy, and facilitating extensive plastic deformation by slowing down the progression of the cracks.

4. Conclusions

Three types of bioinspired architectures have been successfully fabricated here through laser remelting technology. Among them, bioinspired HEA with brick-and-mortar architecture shows an excellent yield strength of ~ 520 MPa with a large uniform elongation of $\sim 35\%$. The synergistic strengthening is mainly attributed to the hetero-deformation of the intricate bioinspired architecture. The intricate bioinspired architecture allows for an effective stress transfer within and between different regions, preventing the premature cracking of the hard regions. The microcracks are strictly arrested at the interfaces of the soft and hard regions, consuming

significant mechanical energy and effectively prolonging the plasticity. Our findings provide a feasible technical route to fabricate biomimetic architecture metal materials and open up a new frontier for the development of high-performance HEAs through biomimetic architectures design.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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