Supplementary Information for

**Non-Hookean large elastic deformation in bulk crystalline metals**

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Supplementary Discussion

1. On the deviation from Hooke’s law

The deviation from Hooke’s law within large elastic deformation may have been experimentally observed in nanowires or whiskers due to lattice anharmonicity owing to the large deviations in interatomic separation from the equilibrium values\(^1\), but it is not a frequent case for bulk crystalline metals in experiments. The linear elastic response of a crystalline material is represented by a quadratic term in the interatomic potential energy; however, nonlinear elasticity requires higher order terms\(^2\). The nonlinear large tensile elastic response in the present bulk Cu–Al–Mn single-crystalline alloy would suggest a high-order elastic behaviour, in which the stress–strain relationship for uniaxial loading can be expressed as follows\(^3\):

\[
\sigma_T = E\varepsilon_T + D\varepsilon_T^2 + \cdots
\]  

(1)

where \(\sigma_T\) is the true stress, which is the applied force \(F\) divided by the actual cross-sectional area \(A\) of the tensile specimen at that load, \(\sigma_T = \frac{F}{A}\); \(\varepsilon_T\) is the true strain, which is equal to the natural log of the quotient of current length \(L\) over the original length \(L_0\), \(\varepsilon_T = \ln\frac{L}{L_0}\); and \(E\) and \(D\) are the incipient Young’s modulus and third-order modulus, respectively. This relationship is valid for any dimensionality and has been used in evaluating large-strain elastic responses in nanostructures such as Pd nanowires and graphene membrane\(^3,4\). Our data for the tensile true stress–strain curves in the present bulk Cu–Al–Mn alloy shows that a quadratic fit describes the nonlinear response until fracture (Supplementary Fig. 8), where \(E\) and \(D\) are determined to be 23.3 and \(-202.8\) GPa, respectively. The ratio between \(D\) and \(E\) \((b = D / E)\), known as the strain-expanded nonlinearity parameter directly scaling the level of anharmonicity\(^3\), was found to be \(-8.70\). This large magnitude of nonlinearity parameter indicates a strong elastic softening upon tension beyond the linear region.

An elastic response implies the existence of a potential energy that is a function of strain. By considering Poisson’s effect, a relationship between Equation (1) and the strain energy \(U(\varepsilon_T)\) related to strain of a crystalline material with Poisson’s ratio \(\nu\) and initial volume \(V_0\) subjected to uniaxial tension can be appropriated as follows\(^3,5\):

\[
\frac{U(\varepsilon_T)}{V_0} = E\left[\frac{1}{2}\varepsilon_T^2 + \frac{1}{3}(1 - 2\nu + b)\varepsilon_T^3\right]
\]  

(2)
where the second-order term is the harmonic term, the third-order term represents the anharmonic contribution. Subsequently, a dimensionless expression for the fractional anharmonic contribution to the strain energy density as function of $\varepsilon_T$ can be expressed as $\frac{2}{3}(1 - 2v + b)\varepsilon_T$. Using $v = 0.4665$ (obtained from Fig. 3), this value would be $-6\%$ and $-25\%$ for tensile true strains of $1\%$ and $4.3\%$, respectively, in the present case (Supplementary Fig. 9a), indicating that lattice anharmonicity plays a substantial role for the strong elastic softening upon uniaxial tension particularly at a large-strain deformation.

Following Milstein’s work, the following relation is obtained as a lattice stability criterion for a cubic crystal:

$$K \equiv \frac{\partial^2 U(\varepsilon_T)}{\partial \varepsilon_T^2} = E[1 + 2(1 - 2v + b)\varepsilon_T] > 0$$ (3)

The $\beta$-BCC lattice becomes unstable to the strain at which the $K$ vanishes. Supplementary Fig. 9b plots the strain energy density $\frac{U}{V_0}$ and force constant $K$ as a function of tensile true strain for the present alloy upon $<100>$ tension. The ideal elastic strain limit can be then estimated to be $5.79\%$ at a strength of $669$ MPa (true strain and true stress). The experimental results of $639$ MPa (true stress) and $4.47\%$ (true strain) greatly approach the ideal values, putting the present Cu–Al–Mn alloy into the “ultrastrength materials” catalogue. Here, the term “ultrastrength” was coined in 2009 to describe material components sustaining sample-wide stress level at a significant fraction (>1/10th) of its ideal strength or $1\%$ elastic strain pervasively.

By using Equation (2), the strain energy density $\frac{U}{V_0}$ as a function of true strain in both tension and compression is plotted in Supplementary Fig. 9c. It is seen that there is an asymmetry in strain energy density between tension and compression because of the lattice anharmonicity.

### 2. Key factors for achieving large tensile elastic deformation

Supplementary Fig.10 compares the strain energy landscape of the present Cu–Al–Mn alloy to that of typical bulk BCC-structured metallic elements for uniaxially stretching along the $<100>$ direction, including Fe, W, Mo and Nb. One can see that the increase in strain energy due to straining is far smaller in the present Cu–Al–Mn alloy, indicating that a smaller applied stress could induce a large elastic strain within the lattice stability region. Incidentally, this mild
strain energy landscape is in consistent with the results of Heusler Cu$_2$MnAl crystals obtained by first-principles calculations$^{12,13}$, which indicates that the crystal could distort easily due to intrinsic weak interatomic bonds. The mild characteristics in energy landscape can also reflect a low value of $C'$ modulus and a low Young’s modulus in cubic crystals$^{14}$.

Nevertheless, the inhibition of plastic deformation is a key issue for achieving a high yield strength. It is traditionally considered that metals showing a low Young’s modulus have a low strength, such as Sn alloys, Al alloys, and Mg alloys. Typically, for example, in $\beta$-Sn or its alloys, a low $C'$ modulus or a low Young’s modulus may be identified, indicating a mild energy landscape on straining, yet their elastic strain is smaller than 0.2% due to the very small yield strength related to the occurrence of dislocation-stimulated plastic deformation$^{15}$. However, being different from those conventional soft metals, the present Cu–Al–Mn alloy is featured by its long-range ordered atomic structure (ordered BCC structure, L2$_1$)$^{16}$, which should give rise to a high resistance to dislocation creation or movement for plastic deformation due to ordering strengthening effects$^{17}$. Ordering strengthening can be mainly considered to be related to the antiphase boundary (APB) energy. Because the creation of superlattice dislocations in ordered structures will form an APB interface, which means that the critical stress to generate and move the dislocations should be larger due to the involvement of APB energy. The high APB energy (503 mJ m$^{-2}$) featured in Cu$_2$MnAl system indicates a high yield strength to plastic deformation$^{18}$. The reported experimental yield strength for a $<100>$ Cu$_2$MnAl single crystal uniaxially deformed at room temperature is about 680 MPa$^{19}$. Incidentally, this value matches the intrinsic strength of 669 MPa in the present alloy. This matching between actual strength and intrinsic strength means that the present Cu–Al–Mn crystal can actually be distorted to a large extent to achieve a large elastic strain without premature involvement of dislocation-stimulated plastic deformation. Some other factors, such as antiphase domain size and quench-in vacancies (Supplementary Fig.12), may also contribute to the ordering strengthening effect$^{17}$, which represent a future research topic for the present Cu–Al–Mn alloy. Hence, large elastic deformation can be obtained in bulk BCC crystals with low resistance to distortion (this can be reflected by a low shear modulus $C'$ or a low Young’s modulus) and a long-range ordered atomic structure for high yield strength.

3. On the difference between elasticity and pseudoelasticity
Reversible deformations caused by mechanical stresses in crystalline metals can be due to a true elasticity or a pseudoelasticity. A true elastic behaviour in crystalline metals is a macroscopic manifestation of atomic bonding and thus a result of bonds against extension or compression and distortion. The elastic strain should then correspond to the reversible lattice strain in the single phase. Being different from a true elasticity, pseudoelasticity can be attributed to several other mechanisms, such as the stress-induced martensitic transformation (also known as superelasticity in shape memory alloys)\(^{20,21}\), the twinning-untwining behaviour\(^{22}\), or the reversible glide of dislocations (in rare cases)\(^{23,24}\). The strain in pseudoelasticity is actually inelastic; however, it is reversible. A true elasticity is noticeable for its features such as the zero stress hysteresis and the monotonicity in stress–strain relations.

Considering the more possible confuses between a true elasticity and a pseudoelasticity induced by stress-induced martensitic transformation (superelasticity), here the evidence for distinguishing them is presented.

- **Stress–strain characteristics**: In superelasticity, the stress-strain curve is usually highly nonlinear with a hysteresis loop (e.g., Supplementary Fig. 13a), because stress-induced martensitic transformation usually occurs in an avalanche-like manner, leading to strain burst and large energy dissipation\(^{20}\). For some alloys exhibiting confined stress-induced martensitic transformation or continuous phase transformation due to defects, the stress hysteresis can be greatly reduced; however, the inflection points in the stress–strain curve would indicate the presence of a weak first-order phase transition\(^{25,26,27}\). For a true elasticity, since there is no occurrence of first-order phase transition, the stress–strain relation is monotonic without any stress hysteresis (Fig. 1a).

- **Microstructure characteristics**: In superelasticity, stress-induced martensitic transformation occurs with the creation of interface between parent phases and martensite phases, which presents the surface relief in the specimen\(^{21}\). In a true elasticity, the deformation is homogeneous across the single phase; therefore, no surface relief or trace appears (Fig. 2a).

- **Phase identification characteristics**: In superelasticity, due to the transformation from parent phase to martensite phase with a different crystal structure, there should be additional peaks or lines that appear in the X-ray or neutron diffraction patterns. Upon the deformation processing, the intensity for the peaks or lines of martensite phases increases whereas that of parent phase decreases. In a true elasticity, the single-phase nature ensures that no additional
peaks or lines appear. The positions of peaks or lines shift continuously due to the lattice parameter change of the single phase during elastic deformation, whereas the intensities remain almost constant (Fig. 3).

- Thermal characteristics: In superelasticity, so-called elastocaloric effect occurs due to the entropy change between parent and martensite phases. In most cases, the temperature of the specimen greatly increases (by ~10 K in Cu-based shape memory alloys) upon adiabatic loading due to the transformation from parent phase to martensite phase, whereas it decreases upon adiabatic unloading due to reverse phase transformation\(^28\). In a true elasticity, a thermoelastic effect occurs due to the pure volumetric deformation of a single phase. Generally, upon uniaxial adiabatic loading, elastic tension will cause a decrease (usually of less than 1 K) in the specimen temperature, and elastic compression will cause an increase in the specimen temperature (Supplementary Fig. 7)\(^29\).

4. On the relationship to pretransitional phenomena in martensitic transformation

In shape memory crystalline alloys, there are some studies reporting a drastic reduction of $C'$ modulus upon cooling of presumably homogeneous parent phase towards the martensitic transformation\(^30\), known as pretransitional phenomena. The significant softening of certain acoustic phonons has also been reported in the cooling process of a parent phase towards martensitic transformation\(^30\). We performed tensile test on the present Cu–Al–Mn alloy at lower temperatures by using another near-\(<100>\) single crystal, and stress-induced martensitic transformation occurs due to the reduced relative lattice stability upon cooling (Supplementary Fig. 13a). It is seen in Supplementary Fig. 13b that the incipient Young’s modulus of the parent phase is almost constant at various temperatures and that drastic change due to pretransitional softening behaviours is not detected even before stress-induced martensitic transformation. This demonstrates that the low Young’s modulus and the huge elastic deformation in the present alloy originate intrinsically in the parent phase, but they are not directly related to the pretransitional phenomena.

5. Elastic responses on compression and bending
Supplementary Fig. 14a shows the engineering stress–strain curves in compression of the present <100> Cu–Al–Mn single crystal. It is seen that the Young’s modulus increases gradually with the increase in stress (elastic stiffening), yet a large elastic strain of over 1.8% can still be reached at an applied engineering stress of 600 MPa. The instantaneous Young’s modulus increases from ~24 GPa at a stress of 20 MPa to ~40 GPa at a stress of 600 MPa, as shown in Supplementary Fig. 14b. The asymmetric elastic behaviour, i.e., the elastic stiffening in compression and elastic softening in tension, is a result of the lattice anharmonicity described in Supplementary Fig. 9c.

Supplementary Fig. 15a shows the force–deflection curves in bending of the present <100> Cu–Al–Mn single crystal. It is seen that the crystal could be bent to a large deflection and then spring back to its original shape after unloading, being different from the bending response of Mo sheets undergoing plastic deformation, as also indicated by Supplementary Fig. 15 b and c. Note that the drop in force on unloading from large deflections in the force–deflection curves should be caused by drift of contact points between specimen and supports. At a deflection of 1.2 mm, the flexural strain \( \varepsilon_{\text{flex}} \) in the outer surface can be roughly estimated to be 2.16% using the following equation\(^3\):

\[
\varepsilon_{\text{flex}} = \frac{6\delta t}{L^2}
\]  

where \( \delta \) (= 1.2 mm) is the deflection of the center of the specimen, \( t \) (= 0.30 mm) is the specimen thickness and \( L \) (= 10 mm) is the support span. Further, it is seen from Supplementary Fig. 15a that the Cu–Al–Mn single crystal is much less stiff than Mo. The bending modulus \( E_{\text{bend}} \) of the present <100> Cu–Al–Mn single crystal can be calculated using the following equation\(^3\):

\[
E_{\text{bend}} = \frac{L^3 m}{4bt}
\]

where \( m \) (= 14.86 N mm\(^{-1}\)) is the slope of the initial straight-line portion of the force–deflection curve and \( b \) (= 3.86 mm) is the width of the specimen. The calculated bending modulus is small to be 32.1 GPa, being slightly different from the value of tensile Young’s modulus, which is due to the different deformation mode between bending and tension.
Supplementary Figures

Supplementary Fig. 1 Electrical resistivity as a function of temperature. There is no abrupt change in electrical resistivity upon cooling, indicating a stable β phase.

Supplementary Fig. 2 Stress–strain responses for tensile deformation to fracture. Engineering stress–strain curves up to fracture by incremental tensile loading–unloading tests at room temperature.
Supplementary Fig. 3 Orientation dependence of the incipient Young’s modulus. The values are calculated using the experimentally determined elastic constants listed in Table 1. The values along the <001>, <011>, and <111> directions are 23.2, 69.2, and 209.1 GPa, respectively.

Supplementary Fig. 4 Stability of the large elastic deformation. The engineering strain is plotted as a function of time at several constant holding stresses. The elastic strain energy density can be stored without relaxation over a significant period.
Supplementary Fig. 5 In situ microstructure observations inside a transmission electron microscope. The bright-field images in front of a crack tip and corresponding electron diffraction patterns at different nominal strain states during in situ tensile deformation at room temperature are presented. There is no phase transformation detected, as known from the invariance of electron diffraction patterns.
Supplementary Fig. 6 The observed macroscopic engineering stress–strain curves during the neutron diffraction measurement. The red and blue dots represent the corresponding {400} d-spacing strains in the direction of loading and unloading, respectively.

Supplementary Fig. 7 Thermoeelastic effect due to the elastic tensile deformation. The specimen temperature was plotted as a function of time during tensile loading and fast unloading at room temperature with a maximum stress of 550 MPa. The specimen temperature increases by about 0.36 K during the adiabatically unloading process due to the thermoelastic effect induced by a true elastic deformation.

Supplementary Fig. 8 True stress–strain curves. The true stress–strain curves of loading to fracture and the fitted curves.
Supplementary Fig. 9 **Energy landscape related to strain.** (a) The strain energy $U$ as a function of tensile true strain. The hatched area represents the anharmonic contribution to the strain energy. (b) The strain energy density $\frac{U}{V_0}$ and force constant $K$ as a function of tensile true strain. (c) The strain energy density $\frac{U}{V_0}$ as a function of true strain in both tension and compression.
Supplementary Fig. 10 Comparison of energy landscape among the present Cu–Al–Mn and typical BCC elements. The data points for BCC elements were obtained by first-principles calculations\textsuperscript{9,10,11}.

Supplementary Fig. 11 Anisotropic elastic responses. Anisotropic elastic properties during loading and unloading in tension on single crystals with an applied engineering stress of 550 MPa. The inset shows the crystal orientation of the specimen, which is parallel to the uniaxial loading direction. The dashed lines indicate the Young’s modulus calculated using elastic constants in the $<001>$, $<101>$, and $<111>$ directions.
Supplementary Fig. 12 Microstructures at the nanoscale. Dark-filed TEM image taken from the (111)_{L2₁} superlattice reflection and the selected area electron diffraction pattern obtained with a [011] incident zone axis for the present Cu–Al–Mn alloy. The average size of ordered domains is about 8 nm in diameter.

Supplementary Fig. 13 Tensile deformation behaviours at lower temperatures. (a) Tensile stress–strain curves at lower temperatures. Stress-induced martensitic transformation (SIMT) occurs at lower temperatures due to the reduced lattice stability of BCC phase. (b) The incipient Young’s modulus as a function of testing temperature.
Supplementary Fig. 14 Elastic responses on compression. (a) Compressive engineering stress–strain curves of a near-<100> single crystal at room temperature with loading to 600 MPa and unloading to 20 MPa. The red dashed line is a guide representing linear elastic response. (b) The instantaneous Young’s modulus as a function of engineering strain derived from the compressive loading engineering stress–strain curves.

Supplementary Fig. 15 Elastic responses on bending. (a) Force–deflection curves obtained during the bending loading–unloading tests for a <100> Cu–Al–Mn single crystal, the inset shows the crystal orientation along the longitudinal direction of the specimen. The bending force–deflection curves of a Mo sheet specimen easily undergoing plastic deformation are plotted as dashed lines for comparison. The photographs captured during the bending tests for (b) the present Cu–Al–Mn single crystal and (c) the Mo sheet.
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