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## Deformation behavior of high-entropy oxide (Mg,Co,Ni,Cu,Zn)O under extreme compression HPSTAR 1498-2022

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ABSTRACT

Following the discovery of high-entropy alloys, high-entropy oxides have gained considerable interest due to their unconventional structural characteristics and versatile functional properties for promising applications. Via synchrotron radial x-ray diffraction in a diamond anvil cell, the mechanical strength and deformation behavior of a typic high-entropy oxide (Mg,Co,Ni,Cu,Zn)O with a rock-salt structure under extreme compression has been investigated *in situ*. This compound in a polycrystalline state shows a large elastic anisotropy at the initial compression stage and then gradually becomes isotropic at around 21.4 GPa, similar with the behavior of MgO. Based on the lattice strain order conversion and texture evolution under compression, a dominant slip system transition from  $\{100\} < 011 >$  to  $\{110\} < 1-10 >$  is proposed in this high-entropy oxide. This work deepens our understanding on the role of chemical disorder in the mechanical properties of entropy-stabilized oxides, which would be indispensable to the design of advanced structural ceramics with optimal strength-to-ductility ratio.

With the concept of "high entropy" alloys firstly proposed by Yeh et al. [1] and Cantor et al. [2] in 2004, there has been a great effort to investigate and characterize systems with 5 or more components. The equal or near-equal atomic ratio in these compositions maximize the configurational entropy and thus stabilize complex compositions in a single lattice. The boosting of relevant research has been extremely beneficial for the materials community as it has led to the development of many new systems with targeted applications. More recently, besides alloys, the "high entropy" strategy has been successfully extended to oxides and other ceramics like nitrides and carbides [3–5].

In 2015, Rost et al. firstly demonstrated the entropy stabilization in oxides with the synthesis of a complex composition of  $(Mg_{0.2}Co_{0.2}$ . Ni<sub>0.2</sub>Cu<sub>0.2</sub>Zn<sub>0.2</sub>)O in a single rock-salt structure [6]. Following this work, various types of high-entropy oxides (HEOs) have been reported, including fluorite type [7], perovskite type [8], spinel type [9], bixbyite type [10], and amorphous type [11]. These systems were soon proven to

be exhibiting tremendous potential in various applications, including thermo-insulators [12], catalysts [13], batteries [14] and wear-resistant and corrosion-resistant coating [15]. As the investigation of HEOs is still at the early stage, many material characteristics still remain to be explored, especially the mechanical properties, including the elastic and plastic deformation properties under applied stresses. (Mg,Co,Ni,Cu,Zn) O high-entropy ceramics synthesized by field-assisted sintering technology showed a trade-off between densification and grain growth with a maximum bending strength of 323 MPa and elastic modulus of 108 GPa after densification at 900 °C [16]. High pressure experiments on the same HEO gave bulk modulus value of 187.7 GPa and 176.1 GPa for bulk sample [17] and nano crystals [18], respectively. Also, this HEO has been proved to be mechanically stable up to  $\sim 50$  GPa [17]. Another HEO,  $(Ce_{0.2}La_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})O_{2-\delta}$  demonstrated a breakdown of long-range connectivity of lattice and amorphization with applied pressure [19]. However, the strength and plastic deformation behaviors

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of HEOs under stress is still poorly studied.

In this work, via radial x-ray diffraction in a diamond anvil cell (rDAC XRD), the elastic and plastic deformation behavior under compressive stress of a typical HEO, (Mg,Co,Ni,Cu,Zn)O, was investigated in situ under pressure up to 40.1 GPa. The rDAC XRD technique has proven to be quite powerful to understand the deformation behavior of oxides and metals under high pressure [20-22]. Here, x-ray-transparent boron-epoxy gasket inserted into a Kapton support and a panoramic-type diamond anvil cell with large opening was used to allow x-ray going through in radial direction. (Mg,Co,Ni,Cu,Zn)O powder sample was synthesized by solid state reaction at high temperature of 1100 °C in air [17] (Fig. S1) and the high-pressure synchrotron radial x-ray experiment was conducted at beamline BL10XU at Spring-8, Japan [23]. A small fragment of  $\sim 10 \ \mu m$  thick platinum foil was used as a pressure calibrant [24]. No pressure medium wad used to maximum the deviatoric stress on the sample. A pair of diamonds with culet of 300 µm were used to generate high pressure and the sample hole was about 60 µm in diameter. A monochromatic x-ray of wavelength 0.4137 Å was collimated to a beam size of 10  $\mu$ m  $\times$  8  $\mu$ m and focused onto the sample (initial grain size is about a few microns). An imaging plate area detector (IP, Rigaku. Co.) was used to collect x-ray diffraction images. Instrument parameters, such as sample-to-detector distance, beam center, and detector tilt, were calibrated using a CeO<sub>2</sub> standard prior to the experiment. The experiment was carried out at room temperature.

The collected 2D diffraction images were processed by the software Dioptas [25] to get the integrated diffraction patterns. Fig. 1(a) shows all the diffraction patterns obtained during both compression and decompression. Selected unrolled diffraction patterns are presented in Fig. 1 (b). No new diffraction lines appear up to the highest pressure 40.1 GPa reached in this experiment, indicating no phase transition in this pressure range. This is consistent with previous work that this HEO is stable up to ~ 50 GPa [17]. Actually, most relevant single-metal oxides are also quite stable in this pressure range, except that the hexagonal ZnO would transfer to the cubic structure at ~ 9 GPa, and cubic CoO transfers to rhombohedral structure at ~ 42 GPa [26–29]. It should be noted that the splitting of (200) and (311) peaks above 2.8 GPa in Fig. 1(a) is due to the large lattice strain induced by the differential stress, which is a reasonable phenomenon for uniaxial compression in rDAC experiment, while (200) and (311) actually keep to be single diffraction lines up to

40.1 GPa as shown in Fig. 1(b). The larger the differential stress, the stronger the lattice strain, and thereafter a higher curvature degree of the diffraction lines. Lattice strains show anisotropic pressure dependent behavior, judging from the curvature behavior of different diffraction lines. As seen in Fig. 1(b), diffraction line of (200) is more curved than (111) at 2.8 GPa. As pressure increases, (111) line becomes more and more curved while (200) gradually turns to be straighter. To demonstrate this phenomenon clearly, we extracted the d-spacing values of these two diffraction planes at different azimuth angles. Results at three representative pressure points are shown in Fig. 1(c). At 2.8 GPa, the d-spacing of (200) displays much larger variations with azimuth angle, as compared with that of (111). They become comparable at 14.9 GPa. Finally, (111) plane shows larger d-spacing variation than (200) plane.

The variation of intensity on each diffraction line as a function of azimuth angle represents the lattice preferred orientation, or texture. Obvious texture is developed from 2.8 GPa and then becomes stronger with further compression. At 14.9 GPa, it is easy to find intensity maxima at the compression direction (azimuth angle 0, 180, and 360) for diffraction (220). However, from 21.4 GPa, the intensity at compression direction for (220) begins to decrease and no maxima can be seen at 40.1 GPa (see Fig. S2 in supplementary materials), indicating that texture transformation might happen under compression. We also found that the texture obtained at 40.1 GPa can be reserved to ambient condition.

Diffraction images were imported to MAUD software to get quantitatively analysis by Rietveld refinement [30,31] (Fig. S3). Information including cell parameters, microstrain, grain size, lattice strain, and texture were extracted from the refinement results. The data analysis method used here was same as that in our previous work [20,21,32]. Fig. 2(a) and (b) show the lattice parameters and the unit-cell volume of this HEO as a function of pressure, respectively. The volume with pressure is fitted with the Birch-Murnaghan equation of state, obtaining a bulk modulus of  $189 \pm 17$  GPa or  $210 \pm 5$  GPa with a fitted  $K_0$  =5.3  $\pm 1.1$  or a fixed  $K_0$  =4, respectively. This value is comparable or slightly higher than the bulk modulus obtained under hydrostatic compression [17,18]. This is due to the fact that the nonhydrostaticity usually leads to an over estimation of bulk modulus as observed in other materials [20, 33]. When compare with the relevant single-metal oxides, we found that the bulk modulus of this HEO compound was comparable with that of



**Fig. 1.** X-ray diffraction patterns of (Mg,Co,Ni,Cu,Zn)O under pressure up to 40.1 GPa. (a) Integrated X-ray diffraction patterns upon compression and decompression. Black stars \* indicate diffraction peaks from Pt. (b) Selected unrolled diffraction patterns. Green arrows at the left side indicate the compression direction. (c) *d*-spacing as a function of azimuth angle of (111) and (200) at different pressure points.



Fig. 2. Refinement results of (Mg,Co,Ni,Cu,Zn)O at high pressure. (a) Lattice parameters, (b) unit-cell volume, (c) lattice strain, and (d) differential stress of this HEO as a function of pressure. Solid lines in (b) are EOS fitting results. The dotted lines in (c) are guide of eyes.

the rhombohedral-distorted NiO and the cubic-type CoO, but much higher than that of the other compounds [26–29].

Lattice strain Q(hkl) was obtained using the "Radial Diffraction in the DAC" strain mode in MAUD, and results are shown in Fig. 2(c). At the initial compression stage, Q(200) increases rapidly and shows a value of 0.01104 at 2.8 GPa, which is much larger than that of Q(111) (0.00452) and Q(220) (0.00688). Above 6.6 GPa, the value of Q(200) begins to decrease while Q(111) keps gradually increasing and Q(220) stays almost stable. From 21.4 GPa, Q(111) becomes the largest and reaches a value of 0.01034 at 40.1 GPa. Q(200) finally decreases to be the smallest with a value of 0.00763 at 40.1 GPa.

According to the lattice strain theory developed by Singh et al. [34, 35], we have 6Q(hkl) = t(hkl)/G, where t(hkl) is the differential stress and *G* is the aggregate shear modulus of the polycrystalline sample. The

shear modulus at ambient condition for non-magnetic (Mg,Co,Ni,Cu,Zn) O has been calculated to be 88.6 GPa [36]. Its pressure derivation is still unknown and we assume that it has the same trend with MgO [37]. Then, the differential stress of this HEO under high pressure was calculated and results are shown in Fig. 2(d). It is clear that t(200) increases much faster than the others below 10 GPa and then it enters into a plateau with a value ~ 7.0 GPa up to 40.1 GPa. On the contrary, the value of t(111) keeps growing with pressure during the whole compression process and reaches 9.4 GPa at the highest pressure 40.1 GPa. The value and increase trend of t(220) with pressure is in between of t(200) and t(111). It becomes stable above pressure of 14.9 GPa and almost represents the average strength of the specimen.

The texture information was obtained by imposing axial symmetry about the compression direction and represented in inverse pole figure



Fig. 3. Texture evolution of (Mg,Co,Ni,Cu,Zn)O under pressure. Inverse pole figures of the compression direction are shown up to 40.1 GPa and after pressure release. The abbreviation *dp* indicates decompression. Pole densities are measured in multiples of a random distribution (mrd). For each IPF, the minimum and maximum pole densities are shown. Equal area projections.

(IPF) (Fig. 3). From 2.8 GPa, clear texture is shown with a maximum at 001 (m.r.d: 3.85) in the IPF, which means 001 plane is oriented perpendicular to the compression direction. With the increase of pressure, this texture also becomes stronger with an increasing m.r.d value. At 8.4 GPa, another weaker maximum appears at 110. But it does not last long and disappears from 21.4 GPa. The maximum pole density also decreases around 25% at this pressure point. The 001-texture lasts to 40.1 GPa and can be retained when the pressure is released.

The maximum uniaxial stress supported by a material is defined by the yield strength. So, t represents the lower end of the material yield strength under pressure. As compared with previous strength results of MgO [38,39] and MgFeO [40] (Fig. 4(a)), t of this polycrystalline HEO shows a similar pressure dependence, which increases rapidly with pressure and then saturates. Texture formed in the sample can affect the vield strength of a polycrystalline material. Density of dislocations in the sample increases with pressure and causes hardening which leads to the increase of the yield stress [41]. From texture results, we can see that the maximum pole density of IPFs initially increases dramatically with pressure and then begins to decrease and then saturates above 14.9 GPa (Fig. 4(b)). This agrees well with the pressure dependence of the yield strength. Thus, it is reasonable to infer that stress hardening has occurred in this HEO sample, with an increase of dislocation density up to a pressure of about 14.9 GPa. The maximum value of uniaxial stress obtained for this HEO is 8.2 GPa at 40.1 GPa. This value is much higher than that of MgO and MgFeO from Lin et al. [39] and Marquardt and Miyagi [40], but is comparable with the results for MgO obtained by Merkel et al. [38].

The three-independent elastic stiffnesses  $C_{11}$ ,  $C_{12}$ ,  $C_{44}$  have also been obtained and are plotted in Fig. 4(c). Both  $C_{11}$  and  $C_{12}$  display nearly linear pressure dependence while  $C_{11}$  shows a higher increasing speed. However,  $C_{44}$  shows quite abnormal pressure dependence. At 1.2 GPa, it shows a quite large value and then it decreases with pressure and saturates above 2.8 GPa. The increasing trend of  $C_{11}$  and  $C_{12}$  of this HEO with pressure is similar with MgO [37,38,42]. But the abnormal pressure dependence of  $C_{44}$  in the low-pressure range has not been reported in MgO. However, the similar phenomenon has been found in monoclinic CuO, in which  $C_{44}$  first decreases and then increases with pressure with a turning point at 15 GPa [43]. They claimed that this was due to the existence of structural abnormality.

 $C_{44}$  refers to shear on {100} in the  $\langle 0kl \rangle$  direction and  $(C_{11}-C_{12})/2$ represents resistance to shear on  $\{110\}$  in <110> direction. The ratio of these two is known as Zener ratio and can be used to represent the elastic anisotropy of cubic material [44]. Z equals to 1 means elastically isotropy. In radial diffraction experiments, Z under the Reuss approximation can be directly measured without the use of any assumed bulk property [34,45,46]. For this HEO, Z shows a quite large value of ~ 4.9 at 1.2 GPa and then it decreases quickly with pressure and saturats at slightly lower than 1 above 21.4 GPa (Fig. 4(d)). Liu et al. has investigated the elastic properties of NiO in a distorted cubic structure compressed hydrostatically up to 67 GPa and reported a negative elastic anisotropy value (S = $S_{11} - S_{12} - S_{44}/2$ ) through the full pressure range [47]. For MgO, both experimental and theoretical work give a positive elastic anisotropy value, which tends to decrease with pressure, and the material becomes almost elastically isotropic at pressure above  $\sim 20$  GPa [37,38,42]. So, it is clear that this HEO behaves much more like MgO than NiO under high pressure.

The operative slip direction in crystals having the NaCl crystal structure is predominantly <110>. This is also the case for cubic NiO and CoO in which  $\{110\}<110>$  system has been proposed to be the dominant slip system [48,49]. For MgO, although its deformation behavior under high pressure has been thoroughly investigated due to its importance in geophysics, the reported pressure effects are still controversial [50]. Early experiments and theoretical work inferred a dominant  $\{110\}<1-10>$  slip under high pressure[38,51,52].



Fig. 4. Elastic properties of (Mg,Co,Ni,Cu,Zn)O under high pressure. (a) Uniaxial stress component in the HEO measured in this work compared with previous measurements on MgO and MgFeO [38–40]. (b) Texture pole density as a function of pressure. (c) Elastic modules evolution of this HEO under pressure. Solid symbols represent moduli under high pressure calculated assuming no texture in the sample using the Reuss bound. (d) Elastic anisotropy represented by Zener ratio.

Calculations on critical resolved shear stress (CRSS) of single crystal MgO predicted a transition from {110}<1–10> to dominant {100}< 011> slip between 30 and 60 GPa [53]. And high pressure single crystal deformation experiments also showed a change in dominant slip system from to {110}<1–10> to {100}<011> at 23 GPa and 1000 K [54]. However, recent radial diffraction on MgO together with Elasto-ViscoPlastic Self Consistent (EVPSC) modeling results shows a competing of these two slip systems under pressure and {110}<1–10> became the only activate slip system at ~50 GPa [39]. Research on (Mg, Fe)O shows that the ratio of Fe could affect the plastic behavior of (Mg, Fe)O and lead to different lattice strain and texture behavior [55]. However, the microscopic interpretation of this effect remains to be understood.

Via EVPSC modeling, Lin et al. found that a dominant {100}<011> slip system in MgO would cause a 110 texture together with a largest Q (200) and a lowest Q(111), while a dominant  $\{110\}<1-10>$  slip induced a 001 texture with a largest Q(111) and a lowest Q(200) [51]. For this HEO, the much higher Q(200) value in low pressure range suggests the large contribution of the  $\{100\} < 011 >$  slip system at the initial stage of compression, which can be rarely seen for any end-member components. Only one previous deformation-DIA experiment to 8 GPa on MgO shows a similar Q-factor order with Q(200) > Q(200)>Q(200) [56]. However, in that work they did not go to higher pressure and no Q-factor order conversion has been observed. From  ${\sim}5$ GPa, the activity of this slip system begins to decrease while {110}< 1-10> becomes more active, proved by the sudden drop of Q(200). Both slip systems contribute to the texture, and two maxima (100 and 110) could be found on the IPFs. From  $\sim 21.4$  GPa,  $\{110\} < 1-10 >$  becomes the dominant slip system while slip {100}<011> gradually becomes inactive, as same as in the case of MgO [39], which cause the constant growth of Q(111) and the disappear of 110 maximum on the IPF.

In summary, we systematically investigate the deformation behavior of a typical HEO, (Mg,Co,Ni,Cu,Zn)O, under high pressure via radial xray diffraction in a diamond anvil cell. It shows a quite large elastic anisotropy which decreases with pressure and the material becomes almost elastically isotropic above 21.4 GPa. The reverse of trend of *Q* (200) under pressure together with the evolution of texture suggest a transition of dominant slip systems of this HEO under extreme compression. Slip system {100}<011> is dominant at the initial compression stage and then the activity of slip system {110}<1-10> increases and finally becomes dominant under higher pressure.

## **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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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2022.114879.

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