Deformation Mechanisms of Single Crystals and Bicrystals Subjected to Equal-Channel Angular Pressing----Review

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Abstract The deformation mechanisms of various kinds of single crystals and bicrystals during the process of equal channel angular pressing (ECAP) have been paid more attention world wide. This paper reviews the recent progresses in the understanding of the deformation mechanisms of single crystals and bicrystals subjected to one-pass ECAP, and discusses the effect of initial crystallographic orientation and grain boundary on the microstructural evolution of these crystals. Based on those experimental results and analysis, it is suggested that in addition to the shear deformation along the intersection plane (IP) of ECAP die, the shear along the normal of IP also plays an important role in affecting the microstructural evolution and deformation mechanisms of these single crystals and bicrystals.

Introduction

Equal channel angular pressing (ECAP), as one of the major severe plastic deformation (SPD) methods, has been widely used to fabricate ultrafine-grained materials for a decade [1-3]. The mechanism of grain refinement via ECAP has been extensively investigated in numerous materials. In order to get a better understanding of the grain refinement mechanism during ECAP, many researchers selected single crystals and bicrystals as model materials [4-25]. There are several obvious advantages in studying the microstructural evolution during ECAP by employing single crystals and bicrystals. For example, first, one can design the initial orientations of those crystals according to the special purposes; second, due to the simple structures of single crystals and bicrystals, the deformation process of those crystals are relatively easy to be traced after ECAP; third, it is convenient to consider the influence of initial orientations by using single crystals or just study the effect of grain boundary (GB) by employing bicrystals. Since the pioneer works by Wu et al.[4,5] who studied the microstructrural evolution of three copper single crystals with special designed crystallographic orientations during ECAP, Miyamoto et al. [6-9], Furukawa et al. [10-16], Wang et al.[17,18], Grosdidier et al.[19] and Han et al. [20-25] have conducted a series of investigations by using various kinds of single crystals with different orientations and some bicrystals with identical GB.. The topic of these studies includes grain refinement mechanism, twinning behavior, texture evolution, recrystallization mechanism and the relationship between microstructural evolution and deformation model of ECAP etc. The present paper will look back on those studies and have a comparison and emphasis on our previous investigations.

Deformation of single crystals by ECAP

Deformation experiments of single crystals have been attempted in ECAP for a deep insight into the influence of the processing route and crystallographic orientations on the grain refinement mechanism and texture evolution [4-20]. Wang *et al.* [5] first used the special



orientated single crystals to investigate microstructural evolution during ECAP. In their experiments, one of orientation of three single crystals was designed in such a way that the primary slip plane and slip direction are parallel to the theoretical shearing plane and the shearing direction, respectively.

The investigations revealed that, the crystallographic orientation has some effect on the resulted microstructural characteristics especially for the first passage of pressing, and more slip systems were activated, more finer is the initial grain size. Furukawa *et al.* [10-12] investigated the microstructural evolution of an Al single crystal with known orientation during the process of ECAP. They found that the detailed experimental observations are fully consistent with the expectations from crystallographic considerations. Miyamoto *et al.* [6,7] have processed a series of Cu single crystals by the technique of ECAP. They reported that some single crystals with insert direction parallel to [110] still remained as a single orientation after extrusion, while other single crystals have two to three orientations. Those investigators have acquired some interesting results, but several questions still remain, i.e., (a) why some single crystals still remained as a single orientation after severe plastic deformation (SPD)? (b) why the processed single crystal by Furukawa *et al.* [12] has large misorientations after ECAP although it was designed to make one of the slip systems just meeting the macro-scale shear deformation of ECAP? Therefore the microstructural evolutions of single crystals during ECAP still need further investigations.

In order to acquire a clear understanding of the above proposed questions and establish a link between the microstructural evolution and the deformation mode of ECAP, we designed three specially oriented Al single crystals trying to systematically reveal the two issues (i) How does the initial crystallographic orientation affect the refinement process of single crystals? (ii) And how does the deformation mode of ECAP determine the grain refinements of single crystals?



Fig. 1 Schematic illustration of the ECAP coordinates and the current experimental design. (ID for insert direction, TD for transverse direction and ED for extrusion direction)





Fig. 2 Typical SEM-ECC micrographs and the GB misorientation of deformation microstructures for the Al single crystals after one-pass ECAP: (a) and (b) crystal I; (c) and (d) crystal II; (e) and (f) crystal III in ID-ED plane.

Figure 1 demonstrates the schematic of the ECAP die and the corresponding orientations of the three single crystals. These single crystals are named as crystal I, crystal II and crystal III, respectively, as shown in Fig. 1. The insert direction (ID) of crystal I is parallel to [110] and its ED is parallel to $[\overline{1} 11]$. For crystal II, the $(\overline{1} 11)$ is purposely located parallel to the intersection plane (IP) of the ECAP die and the [110] is along the general shear direction on the IP, as demonstrated in Fig. 1. For crystal III, the $(\overline{1} 11)$ is perpendicular to the IP of the ECAP die, while the [110] is vertical to the general shear direction. It is worth pointing out that when a uniaxial compression along the direction of ID is applied, the stress state and deformation between crystals II and III will have no difference in nature, however, in the ECAP deformation, they may behave in quite different manner. The three Al single crystals were processed by ECAP for only one pass, then a systematic microstructural characterization was conducted by using various techniques, and more detailed information is seen in ref [20].

After one-pass ECAP, the three Al single crystals formed totally different microstructrues. Fig. 2 shows the typical scanning electron microscope-electronic channel contrast (SEM-ECC) images and the GB misorientation distribution of those crystals. It can be seen that the refinement results are totally different for the three Al single crystals due to the orientation differences. Crystal I is composed of relatively coarser microstructures than that formed in crystals II and III. The finest microstructures with almost equiaxed grains or subgrains were achieved within crystal II, as shown in Fig. 2(c). While the microstructures for crystal III only consist of a series of ribbons. One can find that those ribbon





Fig. 3 TEM micrographs of deformation microstructures for Al single crystals after one-pass ECAP: (a) crystal I; (b) crystal II; (c) crystal III.

structures make an angle of about 45° with respect to the ED for crystal III, as demonstrated in Fig. 2e. It is found that the misorientation angles of crystal I are almost smaller than 15° , as shown in Fig. 2(b). Therefore, after one-pass ECAP the crystal I may still be regarded as a single crystal if considering 15° as critical value of high-angle GBs. The current results are comparable to the report by Miyamoto *et al.* [6,7], who utilized a series of Cu single crystals with similar orientations. The misorientation angles displayed in Fig. 2(d) have been significantly increased even only by one-pass ECAP for crystal II, which is different from the misorientation changes for crystals I and crystal III, indicating that crystallographic orientation plays a significant role in the microstructural refinement and the dislocation structure evolution. The largest misorientation angle for crystal II is about 50° which is comparable to the results by Furukawa *et al.* [10-12] too. However, Most of the GB misorientation, as displayed in Fig. 2(f). Compared with crystal I, crystal III is more close to a single crystal because most of the GB misorientations are less than 10° . Those results above remarkably demonstrate that the significant effects of crystally crientation on the microstructural refinement and the dislocation structure evolution structure evolution are less than 10° .

The extruded samples were then inspected by transmission electron microscopy (TEM) with the observations taken on the ID-ED plane at the centre part of the billet. Totally different microstructural features were found in those extruded single crystals. After the first pressing, the microstructures in the ID-ED plane of crystal I mainly consist of cell blocks with approximately square shape divided by two sets of dense dislocation walls perpendicular to each other, as shown in Fig. 3(a). The characteristic of the microstructures for crystal II is distinct from the cell block microstructures of crystal I. The microstructures for crystal II mainly consist of two sets of subgrains. The angle between the two directions is about 70°, as demonstrated in Fig. 3(b), indicating that the slip systems on the two main slip planes were activated in crystal II during ECAP. Furukawa *et al.* [10-12] also found the similar types of microstructures in their experiment and labeled as A and B within a single crystal having the same orientation with crystal II, but extruded



in a slightly different ECAP die. However, they considered that the formation process of regions A and B were induced by the crystallographic orientation rotation. The microstructure in crystal III is



Fig. 4 The illustration of the deformation principle during ECAP.

characterized by one set of extended dislocation boundaries and is parallel to one slip plane trace, showing one primary slip plane were dominantly activated during ECAP, as shown in Fig. 3(c).

The results obtained above provide a clear demonstration for the significant influence of the crystallographic orientations on the dislocation structure formation and the misorientation distribution of induced grains and sub-grains in single crystals subjected to ECAP. The different microstructural features for single crystals with different orientations must be induced by the interaction between shear deformation imposed by the ECAP die and the intrinsic slip deformation of those single crystals. Then those results were analyzed by using the concept of shear factor [10]. Based on the analysis, it is suggested that in addition to the main shear deformation along the IP, the shear deformation along the normal direction of IP is also very important.

The specially designed experiments described above obviously indicate that the microstructural features of the three single crystals are well explained by the new suggested deformation mode [20]. Based on the analysis above, we propose the possible deformation mode during ECAP for the ideal case, as shown in Fig. 4. We believe that in addition to along the IP, shear deformation along the normal plane to the IP can also activate the preferential slip system of single crystals during ECAP, at least on micro-scale. This viewpoint is different from the well known 'pure shear theory', i.e., shear deformation only occurs on the IP in the ECAP process [1,2]. In that paper, it is indeed there are some shortcomings such as an initial pole figure was mistaken during review process and did not explicitly point out the shear on the IP. In order to further clarify the deformation mode of ECAP, some Cu bicrystal experiments were conducted and will be introduced in section 4 in this paper.

In summary, crystallographic orientation has a remarkable influence on the dislocation structure evolution of different single crystals during ECAP. According to the deformation process of single crystals, the shear deformation along the normal of IP should also be noticed.

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Deformation twinning in single crystals during ECAP

Slipping and twinning are two major plastic deformation modes in crystalline materials. In general, slipping is often the predominant deformation mode and twinning is extremely difficult to occur in face-centered-cubic (fcc) crystals under conventional loading conditions. In a paper by Miyamoto *et al.*[6], they claimed that deformation twinning was found in Cu single crystals subjected to ECAP for only one pass at room temperature (RT). However, they only showed the metallographical image of some banding structures and no further evidences for deformation twinning. Afterwards, Huang *et al.*[27] have successfully observed deformation twins in coarse grained and ultrafine grained Cu processed by ECAP at RT and low strain rate. But the densities of deformation twins in those experiments is relatively low. In the experiments conducted by Huang *et al.*[27], there is an interesting phenomenon: deformation twins can only nucleate in some special grains with particular crystallographic orientations. This results indicate that deformation twinning maybe more easier in some grains with preferential orientation. Based on those experimental results, it is naturally to put forward a question: whether one can design a single crystal with special crystallographic orientation twinning in ECAP?

The prominent feature of ECAP is that the intense shear strain is imposed along a well-defined plane around the intersection of the input and output channels [1,2]. It means that the plastic deformation of the materials along the other directions will be severely restricted by the channel wall. Therefore, the special deformation mode of ECAP may provide a possibility to effectively suppress the activity of slip systems for some specially oriented single crystals even at low strain rate and at RT. On the other hand, fcc crystals have the definite twinning plane and twinning direction, i.e. $\{111\}$ and $\langle 112 \rangle$, respectively. Then it gives rise to an interesting question: if a twinning system in a fcc single crystal, i.e., (111)[112], is just placed meeting one of the deformation direction ECAP macroscopic shear of [1,2,20], whether press



Fig. 5 Schematic illustration of the specially designed single crystal for twinning.





Fig. 6 (a) and (b) typical SEM-ECC morphology of the shear band formed in copper single crystal after extrusion; (b) illustration of the location of shear bands in the extruded crystal.



Fig. 7 TEM micrographs of the deformation microstructures in copper crystal: (a) the image of the matrix region; (b) the fine strip structures in the shear band; (c) and (d) deformation twins formed during ECAP.

the twinning becomes active or not for the plastic deformation even at low strain rate and at RT? Obviously, the corresponding experimental results will be beneficial for better understanding of the fundamental competition mechanisms between slip and twinning in fcc crystals.

The crystallographic orientation of the Cu single crystal and Al single crystal in the current study was designed with (111) plane perpendicular to the IP of ECAP and with $(\overline{112})$ plane parallel to the IP, as demonstrated in Fig. 5. In this case, the twinning system will acquire the maximum resolved shear stress [20]. Then those single crystals were processed by ECAP for one pass. The

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formed microstructures were observed by both SEM and TEM techniques. SEM-ECC observations shown in Fig. 6 revealed that dense shear bands were formed on the shear flow plane (just the $(\overline{1}10)$ crystallographic plane) of the Cu single crystal after pressing. Those shear bands have a width range from 10 to 50 µm and make an angle of about 20° with respect to the extrusion direction. The area of the shear regions occupies about 10% of the total area. Figs. 6(b) and (c) display a the typical shear band, which consists of two types of microstructures. The region I in Fig. 6(c) stands for the area inside the shear band with very fine banding structures along the shear direction. The region II in Fig. 6(c) represents the area outside the shear bands with coarse ribbon structures, having an interaction angle of about 70° with respect to the shear band. According to the initial orientation, it is easy to calculate that the shear bands were formed approximately aligning along the initial [112] crystallographic direction, as shown in Figs 6(b) and (c). The ribbon structures in the area of matrix are divided by dense dislocation walls with the width of ~500 nm, as demonstrated in Fig. 7(a). The corresponding selected area diffraction (SAD) pattern indicates that those dislocation walls are formed along one of (111) planes. The misorientation angles between those subgrain structures are very small, as proved by the sharp diffraction spots. This region stores high density of dislocations, which is the common feature of those metals subjected to severe plastic deformation [27]. However, the microstructures in the area of shear bands are very fine, consisting of a series of strip structures with the width less than 500 nm, and aligned along the direction of shear bands (Fig. 7(b)). The SAD pattern consists of diffraction rings (Fig. 7(b)), indicating that the misorientation angles between those strips are large. The microstructures in the center of the shear presented deformation distribute band are in Figs. 7(c)and (d), many twins



Fig. 8 Typical SEM-ECC and TEM micrographs of deformation microstructures in Al single crystal after one-pass ECAP: (a) and (b) the formed coarse ribbon structures and shear bands; (c) and (d) the formed subgrains and dislocation structures.

desultorily and discontinuously in this area, as proved by the corresponding SAD pattern. Extensive observations by TEM revealed that the deformation twins in the present crystal can be categorized into three types according to their locations and morphologies and one can find more details in ref [23]. These experimental results demonstrate that Cu single crystal can deform by twinning at a very low strain rate and at RT during ECAP when a proper crystallographic orientation was specially selected with respect to the ECAP die. However, many other experiments also processed a series of Cu single crystals with random orientations at RT and at low strain rate by ECAP or other deformation methods [6, 27-31], only a few of deformation twins were observed. The generation of



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abundant deformation twins in the current Cu single crystal at low strain rate and at RT may be due to the following reasons. Firstly, the special deformation mode of ECAP can exert a considerable constraint on slip systems during extrusion although Cu crystal has many independent slip systems. Secondly, we designed the orientation of the Cu single crystal purposefully to make one of twinning systems just meet one of the macroscopic shear deformation of ECAP, leading to a higher level of resolved shear stress on the twinning system than those on most slip systems, which can also assist the constraint process of slip during extrusion. In addition, the microstructures were severely refined during ECAP, which can also apply a confining effect on the dislocation slip activities. Therefore, the slip activities can be suppressed to some extent under this deformation condition, and twinning would become an active deformation mode during extrusion.

Based on the understanding of crystallographic orientation on twinning behavior in Cu single crystal, it is natural to come out of an interesting question: whether one can acquire deformation twins or not in pure Al single crystal deformed at RT and at low strain rate through the special design of crystallographic orientation? Then we cut an Al single crystal having the same orientation with the above Cu single crystal and extruded by ECAP for only one pass. Fig. 8 shows the microstructural morphology of Al single crystal after one-pass ECAP. Two directions of banded structures were formed on the ID-ED plane of the pressed rod, as demonstrated in Fig. 8(a). Those banded structures are all



Fig. 9 HRTEM micrographs showing the formed microtwins and stacking faults in the as-extruded aluminum single crystal. (a) and (b) the HRTEM images acquired by a TEM working at 200kV; (c) and (d) the HRTEM images acquired by a TEM working at 300kV.

with the width of $\sim 4\mu m$, and intersecting an angle of $\sim 70^{\circ}$ between primary and secondary bands. In some other regions of the observed area, very few narrow shear bands were found, as shown in Fig. 8(b). Compared to the dense shear bands formed in Cu single crystal, the shear bands are very few in Al single crystal. After extrusion, many cell-block structures and microbands with the scale of 2 to 3 µm were developed (Figs. 8(c) and (d)). The corresponding SAD pattern indicates that the misorientation among those substructures is small. However, in this scale, there is no any deformation twin in these microbands or cell-block substructures, which is different from the previous observations in Cu single crystals [23]. In order to further reveal the local microstructure information, we observed the Al single crystal by high-resolution TEM. Fig. 9 shows the HR-TEM images acquired from two TEM with different working voltage (200kV-JEM 2010 and 300kV-Tecnai G²F30). HRTEM observation revealed that profuse nanoscale strips with regular shape were



formed after ECAP in the interior of the subgrain, as labeled by the arrowheads in Figs. 9(a) and (b). It is interesting to find that these strips mainly align along two directions with an intersecting angle of $\sim 70^{\circ}$, which is very close to the dihedral angle of the two {111} planes. Further observations on the lattice image clarify that, those strip structures are microtwins or stacking faults with 1-2 atomic layers thick, as demonstrated in Figs. 9(c) and (d). The HR-TEM observations above experimentally provide direct evidences confirming that microtwins or stacking faults really nucleated in Al single crystal deformed at RT and at low strain rate, which is considered impossible previously because the stacking fault energy (SFE) of Al is very high [31-33].

In summary, plastic deformation behaving in a twinning mode is often considered to be extremely difficult in the fcc metals with medium to high SFE due to the prior slip deformation. The present investigation demonstrates a direct evidence of twinning in Cu and Al single crystals with preferential orientations and provides a new understanding on the physical nature of the fundamental plastic deformation mechanisms in various fcc metals.

The deformation modes of bicrystals subjected to ECAP

520

The mechanism of grain refinement via ECAP has been extensively investigated in numerous



materials, including both polycrystals and single crystals [1-23]. However, the investigations by using polycrystals can not well discern the individual influences of numerous GBs and the different orientations of the component grains on the grain refinement mechanism. Therefore, the use of various kinds of single crystals provides a unique opportunity to attain a direct evaluation of the effect of crystallographic orientation on grain refinement [4-23]. However, the studies by using single crystals also have some obvious limitations. For example, it is not clear that what the roles of initial high-angle GBs (HAGBs) are during the process of ECAP because there is no any GB in single crystals. In order to get a better understanding on the effect of both GB and crystallographic orientation on grain refinement, bicrystals with one flat HAGB plane are the ideal model materials. Besides, In order to further investigate the shear deformation mechanisms during ECAP, it is necessary to select bicrystals as model materials because the GBs in the bicrystals can be easily traced during plastic deformation. According to the deformation and evolution of the GBs in the designed bicrystals, one can judge the shear deformation mode of ECAP rather easily.





Fig. 11 The typical SEM-ECC images of the four Cu bicrystals: (a) A-GB-0°; (b) B-GB-45°; (c) C-GB-90°; (d) D-GB-135°.

The present experiments were conducted using bicrystal of high-purity OFHC Cu (99.999%) grown from the melt using the Bridgman method in a horizontal furnace [34]. The initial crystallographic orientations of the bicrystals were determined using the electron backscatter diffraction (EBSD) method. In order to investigate the evolution of GBs in a group of identical bicrystals and the interaction mechanism between shear deformation and GBs during ECAP, the initial position of the GBs in the bicrystals was designed according to the geometrical character of ECAP die. Fig. 10 is the schematic illustration of the GBs in the four Cu bicrystals [24]. The angles between the GBs in the four bicrystals and the extrusion direction (ED) of ECAP die are equal to 0°, 45°, 90° and 135°, respectively; hence the four bicrystals were named as, bicrystal A-GB-0°, bicrystal B-GB-45°, bicrystal C-GB-90° and bicrystal D-GB-135°, respectively.

521





Fig. 12 The typical SEM images of the etched four Cu bicrystals: (a) A-GB-0°; (b) B-GB-45°; (c) C-GB-90°; (d) D-GB-135°.

Figure 11 shows the SEM-ECC images on the ID-ED plane of the four bicrystals in the vicinity of GB after one-pass ECAP. It can be found that the GB in the four bicrystals gets different angles with respected to the ED, as listed in Table 1. Compared to the initial GB, the four bicrystals have been rotated along the anti-clockwise direction for various angles, as listed in Table 1. For the four Cu bicrystals, they have some common features: firstly, GB affected zone (GBAZ) was formed due to the strong effect of GB; secondly, the deformation behavior of the regions far from GB are mainly controlled by the local crystallographic orientations, as shown in Fig. 11. Another striking features formed in those Cu bicrystals is a kinds of special shear bands, as demonstrated in Fig. 12. It can be seen that the GB in the deformed bicrystal A-GB-0° still keeps straight although it has undergone SPD, as shown in Fig. 12(a). Profuse shear bands can be clear observed at the vicinity of GB, as marked in the figure. It should be pointed out that those shear bands did not stop at GB, but still keep a certain distance. The GB in the deformed B-GB-45° bicrystal has been bended due to the strong interaction between the shear bands and the GB. Clear shear bands can be observed in the etched specimen, as marked by the dash lines in Fig.12(b). Obviously, one shear band corresponds to one curvature at the region of the GB and just propagates along the vertical direction of GB in the bicrystal B-GB-45°. Therefore the kinking deformation of the GB in the bicrystal B-GB-45° was induced by the strong shear deformation imposed by ECAP die along the normal direction of IP according to the initial experimental design. For bicrystal C-GB-90°, a series of small shear band traces can be observed at the GB, as shown in Figs. 12(c). Those small shear bands get an angle of \sim 45° with respected to the GB. Due to the shear deformation along the 45° direction, the GB in bicrystal C-GB-90° has been twisted and forms a small step, as displayed in Fig. 12(c). Compared to that formed in the bicrystal B-GB-45°, the steps in the bicrystal C-GB-90° are very small. The SEM observation of the etched sample demonstrates that the bicrystal C-GB-90° has undergone a shear deformation along the 45° direction of the GB. The GB in the deformed bicrystal D-GB-135° also still keeps straight although it has undergone SPD, as shown in Figs. 12(d). Abundant shear bands with the direction approximately parallel to the GB can be seen in Fig. 12(d), indicating that strong shear deformation occurs along the direction of GB. In the vicinity of GB, the etched bicrystal D-GB-135° has very flat surface,



Specimen No.	$\boldsymbol{\theta}_{\!\!\!D}$	$\boldsymbol{\theta}_{F}$	$\boldsymbol{\theta}_{R}$	$\theta_{\rm P}$	$\Delta \theta$	GB state
Bicrystal A-GB-0°	0°	27°	27°	27°	0°	straight
Bicrystal B-GB-45°	45°	53°	8°	45°	8°	curved
Bicrystal C-GB-90°	90°	0°	90°	0°	0°	curved
Bicrystal D-GB-135°	135°	3 0°	75°	18.4°	11.6°	straight

demonstrating that those regions have undergone a relatively homogeneous deformation, which is obviously different from the region far from the GB, as shown in Fig. 12(d). According to the rotation behavior of the GBs during ECAP, one can judge that those shear bands are just induced by the strong shear deformation along the normal direction of IP. Therefore the shear stress along the normal direction of IP also plays an important role in the plastic deformation of Cu bicrystals, which is consistent with the experimental results of Al single crystals as introduced in section 2 in this paper.

In summary, due to the existence of the GB in Cu bicrystals, a special region named as GBAZ were formed in the four Cu bicrystals with different GB orientations after one pass ECAP. From the interaction mechanism between shear bands and GBs in those deformed bicrystals, one can believe that the shear deformation along the normal direction of IP also plays an important role during the process of ECAP.

Conclusions

Based on the experiments conducted by using various single crystals and bicrystals with specially designed orientation and GB direction, we can find that the microstructural evolution process and preferred deformation mechanism have been significantly influenced by the initial crystallographic orientation and the initial GBs. These experimental results also indicate that in addition to the shear deformation along the IP of ECAP die, the shear along the normal of IP also play an important role in affecting the microstructural evolution and deformation mechanism of these single crystals and bicrystals.

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