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Heterogeneous deformation of Cu_2O single crystals during high temperature compression creep*

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Single crystals of Cu_2O in parallelopipedic and cubic geometries have been deformed in high temperature creep. Depending on the compression axis, either 2 or 4 glide planes are observed. Local strain analysis performed by a visioplasticity method permits an estimation of the heterogeneity of deformation and shows clearly the imperfection of the compression test.

1. Introduction

In most studies of compression creep in single crystals little regard has been given to the sample shape after creep. Blum and Ilschner [1] working on high temperature creep of NaCl observed that samples with an initial ratio of the length to the square root of the cross-section larger than two ($l_0/\sqrt{S_0} \geqslant 2$) deformed by glide on two orthogonal planes while cubic samples ($l_0/\sqrt{S_0} = 1$) deformed by glide on four planes. They showed that the $l_0/\sqrt{S_0}$ value does not affect the steady state creep mechanism, and so decided to work on cubic samples. Poirier [2], also working on NaCl high temperature creep, proposed a geometrical construction to explain the barrel-like geometry assumed by parallelepipedic specimens after creep ($l_0/\sqrt{S_0} = 2.4$); he imagined that the deformation occurs by alternating homogeneous glide within two orthogonal bands inclined at 45° to the compression axis. This mechanism implies that the triangle-shaped regions at the top and bottom of the samples [2] are displaced without deformation and that the samples barrel independently of friction on the compression faces. These two studies were performed on samples with the same type of orientation, which in the present study will be referred to as C samples (Fig. 1a).

In the study of Martinez *et al.* [3], Cu_2O samples of another orientation (Fig. 1b) displayed only two glide planes. Samples of this orientation are referred to as CX samples in the present work. The mechanical properties of these two kinds of Cu_2O specimens show many differences; in particular the creep curves are very dissimilar [4]. It is also very interesting to compare the shapes of these two types of sample after creep.

Deformation is not homogeneous inside the samples and most authors have studied the microstructure without knowing the true local strain of the observed regions. This knowledge is very important for the microstructure is obviously not the same everywhere in the case of heterogeneous deformation. We have therefore tried to estimate the heterogeneity of the deformation.

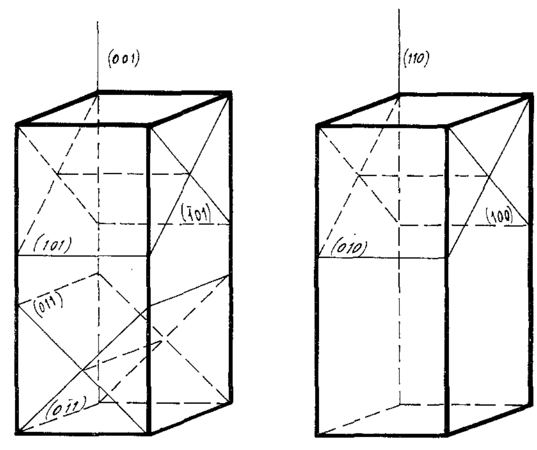


Figure 1 The two specimen types; (a) C, (b) CX.

*Based in part on a thesis to be submitted by T. Bretheau.

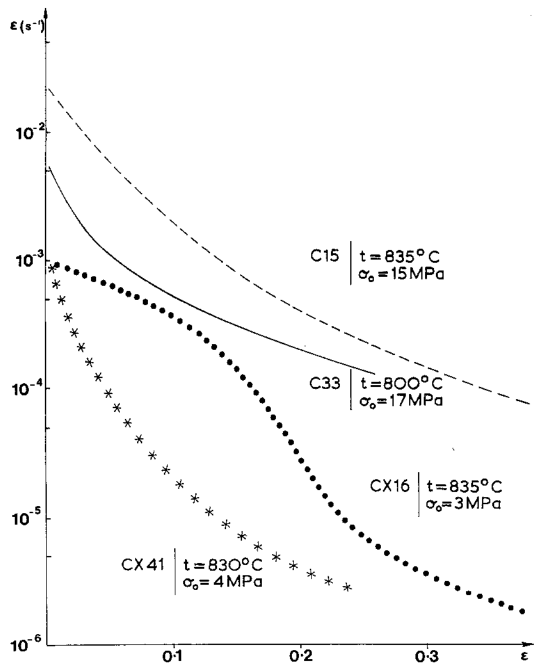


Figure 2 Typical creep curves (strain rate versus true strain) of the two types of specimens: C 15 and CX 16; parallelepipedic samples, C 33 and CX 41; cubic samples.

2. Mechanical tests

The samples were 2.5 mm x 2.5 mm x 6 m parallelepipeds or 3 mm x 3 mm x 3.1 mm cubes. Their preparation has been described elsewhere [4]. CX and C samples were cut. The CX samples (Fig. 1b) were subjected to compression creep along the [1 1 0] axis according to the procedure described by Bretheau *et al.* [4]; there are two {1 0 0} planes inclined at 45° to the compression axis which are subjected to the maximum resolved shear stress. C samples (Fig. 1a) were subjected to compression creep along the [1 0 0] axis; there are four {1 1 0} planes inclined at 45° to the compression axis, all subjected to the maximum resolved shear stress. The only geometrical difference between these two types of samples is the number of potential glide planes. In the case of cubic samples all the glide planes cut one of the compression planes and easy glide was impossible.

Four typical constant load creep curves are shown in Fig. 2 for the two types of parallelepipedic samples and the two types of cubic samples. The principal feature to be noted is the great difference in variation of creep rate with deformation. The parallelepipedic CX crystals exhibit three stages while the parallelepipedic C crystals exhibit only two stages [4]. The CX cubic samples exhibit only two stages (not three) just as the C parallelepipedic samples do. The creep curves of orientation cubic samples are essentially the same as those of C parallelepipedic samples with a shortening of the transient stage. The different creep curves and the creep substructure are described in detail in other publications [4, 5].

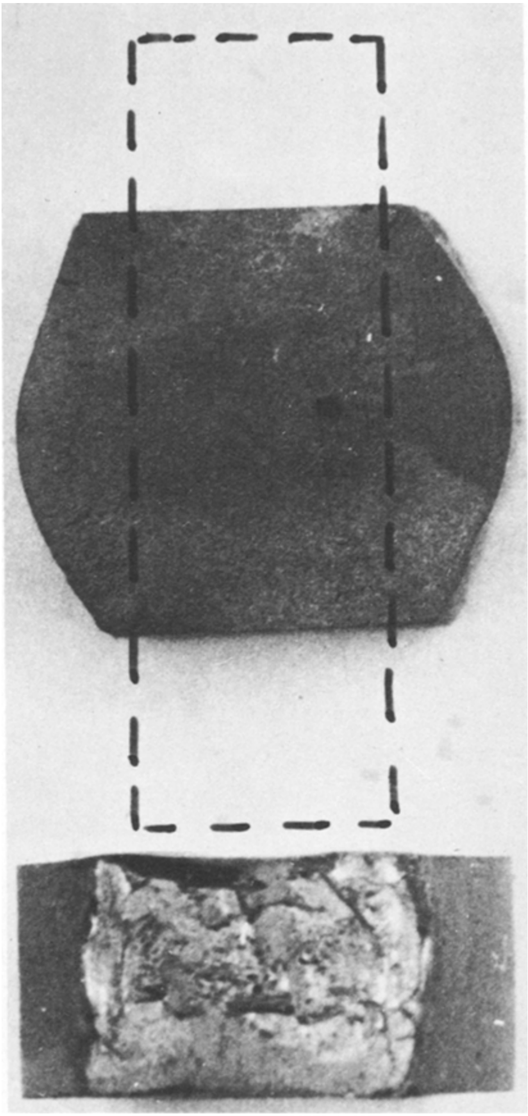


Figure 3 Barrel-like profile of a CX specimen after 50% deformation. The initial profile of the specimen is represented by dotted lines. Before deformation the specimen was 9 mm × 3 mm × 3 mm in order to reinforce the widening of the compression faces and display this effect clearly.

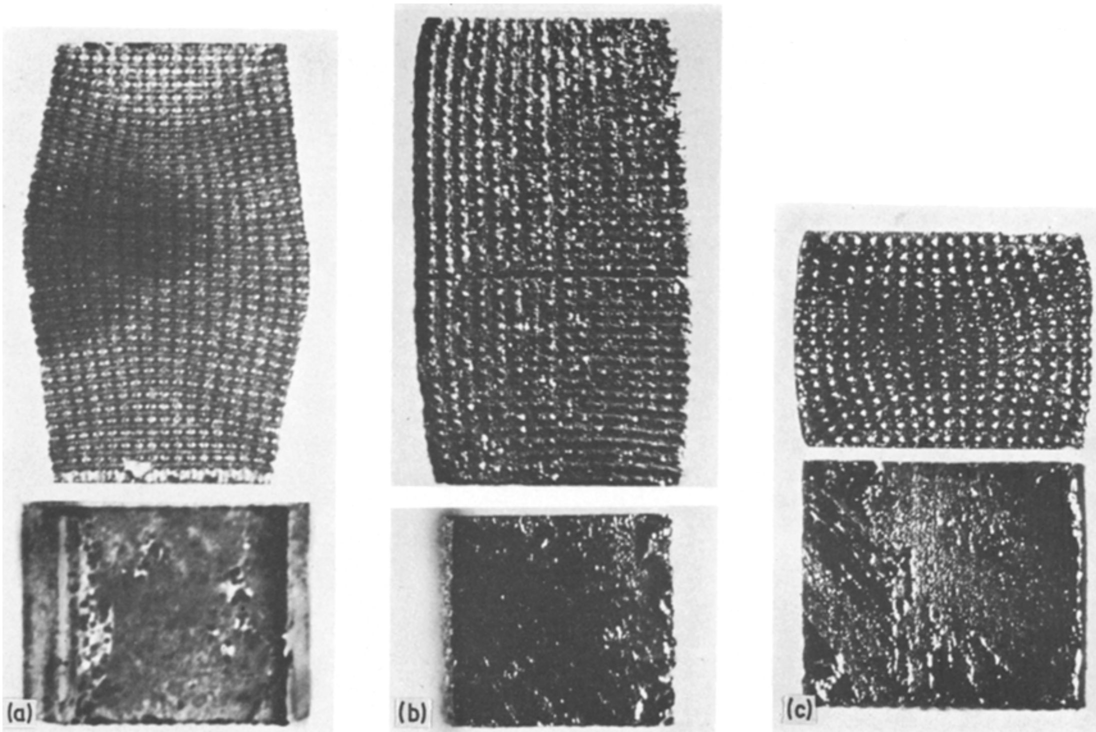


Figure 4 Barrel-like profiles of specimens after 17% deformation; (a) parallelepipedic CX, (b) parallelepipedic C, (c) cubic CX. We can observe the grid patterns used for the strain distribution calculation.

3. Specimen shape after creep

After creep, the two types of specimens are barrel-shaped (Figs. 3 and 4). Actually only two faces barrel while the other two remain plane and parallel. The two barrelled faces are indeed $(\bar{1}10)$ for the CX samples but they cannot be predicted for the C samples. This plane strain can be observed for strains up to 60 to 70% for the CX samples and only up to 40 to 50% for the C samples. The plane strain is the only similarity between CX and C specimens; CX ones exhibit a much more pronounced barrelling than C ones. Furthermore, the top and bottom faces of the CX specimens widen (Fig. 3) while those of the C ones remain square without any area increase.

The CX cubic samples creep in plane strain (Fig. 4c) and the sample ends broaden, but less than in the case of the parallelepipedic CX samples. On the other hand, during the C cubic sample deformation there is no plane strain and we observe a widening of the sample ends, which remain square.

In order to refine our knowledge about the deformation of these samples. We have used an original visioplasticity method to draw up the local true strain distribution.

4. Local strain analysis

4.1. Experimental technique

There is no normal stress on a free surface. If a circular grid pattern is drawn on such a surface, each circle becomes an ellipse after a small deformation (Fig. 5a). The true local strains are given by:

$$\epsilon_1 = \int_\rho^b \frac{dx}{x} = \ln \frac{b}{\rho},$$

$$\epsilon_2 = \int_\rho^a \frac{dx}{x} = \ln \frac{a}{\rho},$$

and ϵ_3. The subscripts 1 and 2 refer to the principal axes of the ellipse and the subscript 3 to the outer perpendicular to the surface (Fig. 5a). a and b are respectively the semi-major and the semi-minor axes of the ellipse and ρ is the initial circle radius.

The material incompressibility assumption dictates;

$$\epsilon_1 + \epsilon_2 + \epsilon_3 = 0.$$

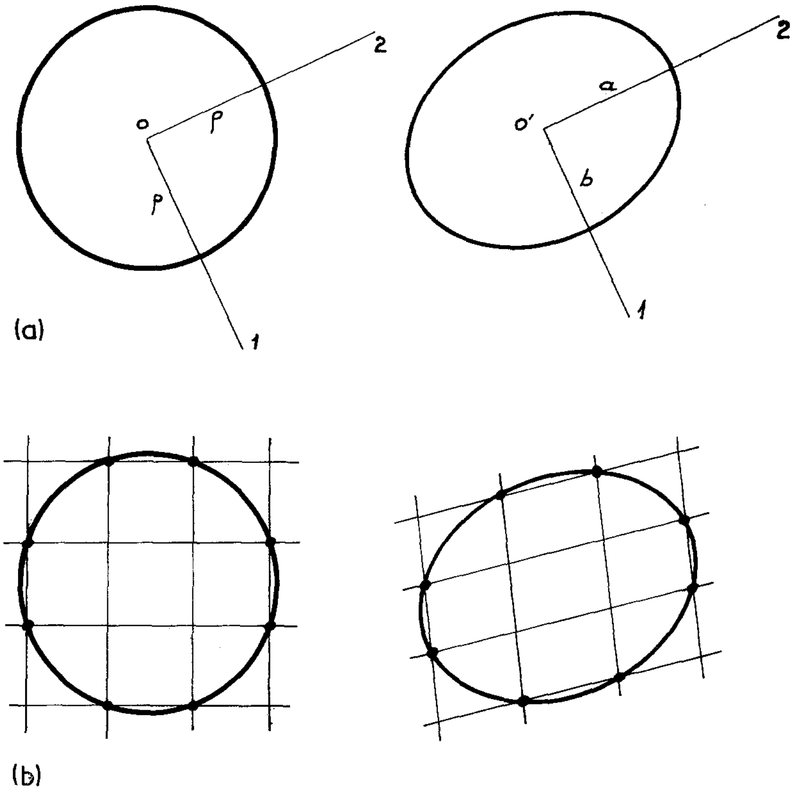


Figure 5 Method used for the calculation of local strains; (a) after deformation a circle becomes an ellipse, (b) drawing of a circle and of the corresponding ellipse, by means of the grid pattern.

If the samples deform in plane strain, i.e. $\epsilon_3 = 0$,

$$\epsilon_1 = -\epsilon_2 .$$

At the present time we cannot make a circular pattern grid on cuprous oxide. We have therefore cut a square pattern grid using a diamond blade on a milling machine. We have obtained a 0.15 mm pitch grid with a maximum depth of 15 μm.

The calculation of the local true strains by a direct method using the square pattern grid is quite difficult. However a very simple relation allows us to use the method discussed previously. Before creep, eight nodes of the square pattern grid define one circle; after creep, these eight nodes define an ellipse (Fig. 5b). We therefore drew the approximate ellipses and measured their major and minor axes. The method described above then gives the local strains.

There are two causes of inaccuracy in this method: (1) Rigorously, a deformed circle is an ellipse only if the strain is very small. This is not the case in our experiments where strains of about 17% were applied; (2) The drawing of the ellipses is not very precise. In fact values of ϵ_1 differing from corresponding values of $(-\epsilon_2)$ were observed; the relative error is always below 20%. We have thus considered a mean local true strain;

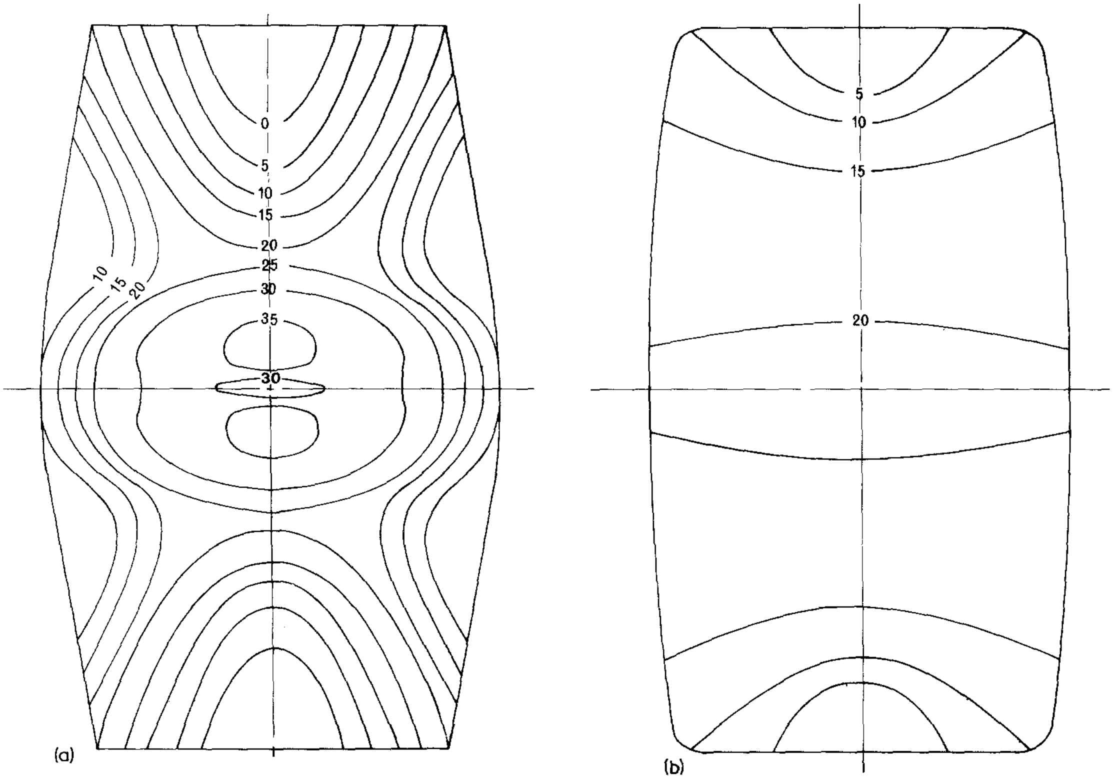
$$\bar{\epsilon} = \frac{\epsilon_1 - \epsilon_2}{2}.$$

No sample was perfectly symmetrical after creep, however data were analysed assuming an ideal symmetrical profile, symmetry being re-established by calculation of the mean strains, taking into account the two symmetry axes. The overlapping of the ellipses was sufficient to give the strain every 0.3 mm. We observed that the values vary continuously from point to point.

4.2. Results

4.2.1. CX samples

4.2.1.1. Parallelepipedic samples CX samples deform in plane strain (Figs. 3 and 4a) with a strong curvature in the middle of the barrelled faces. We suggest a deformation mechanism by alternate homogeneous glide within bands inclined at 45° to the compression axis as did Poirier [2]. Contrary to Poirier, we observe a broadening of the sample ends. The study of the iso-strain lines (Fig. 6a) and the observation of a sample in grazing light (Fig. 7)



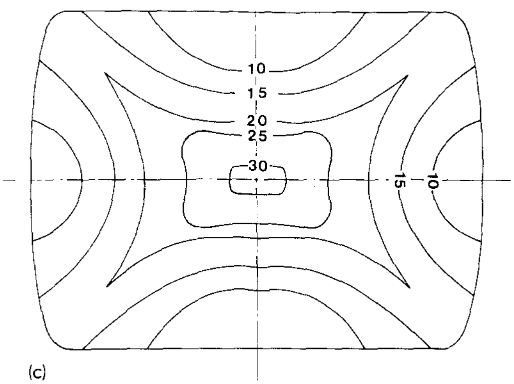


Figure 6 Iso-strain lines after 17% deformation; (a) parallelepipedic CX, (b) parallelepipedic C, (c) cubic CX.

suggest that undeformed triangular regions do exist and that the widening of the specimen ends is due to a concentrated shearing. In the central zone, the strain distribution seems to be quite complex: we observe a relative minimum value of the strain in the centre of the sample between two absolute maxima along the vertical axis and two relative maxima along the horizontal axis. However the method is not accurate enough to resolve that problem in the central zone, so the iso-strain lines were drawn there only as an illustration.

It should be noted that the highest strain is about 35% while the lowest is 0% for a macroscopic true strain of 17%. This shows how heterogeneous the deformation of the CX samples is.

4.2.1.2. Cubic samples The CX cubic samples deform in plane strain. The analysis of the local strains shows (Fig. 6c) that the heterogeneity is

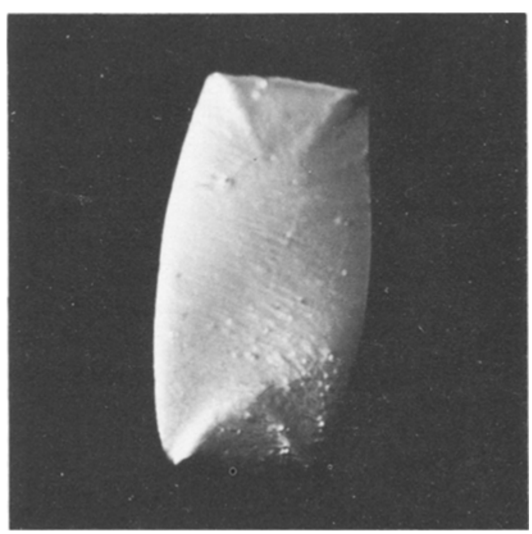


Figure 7 Observation in grazing light of a parallelepipedic CX specimen after 16% deformation.

hardly less important than in parallelepipedic samples.

4.2.2. C samples

4.2.2.1. Parallelepipedic samples As with CX samples only two faces barrel during creep (Fig. 4b) although four glide planes can operate. The profile of the C samples is quite different: barrelling is less pronounced and much more regular, and the ends of the samples do not broaden.

The analysis of the local strains shows fairly good homogeneity of the deformation in the central zone (Fig. 6b). Except for a large strain gradient near the ends, 80% of the sample is 17 ± 3% strained. We cannot observe any zone free of deformation.

4.2.2.2 Cubic samples The C cubic samples do not deform in plane strain: it was impossible to establish the local strain distribution by mean of the method previously described. Nevertheless, the sample shape after creep shows that the deformation is very homogeneous and that the four glide planes operate equally.

5. Discussion

A polycrystal which creeps without friction at the compression faces, deforms homogeneously, but even without friction, a single crystal with a ratio $l_0/\sqrt{S_0} > 1$ barrels because of the anisotropy of the glide systems [2]. Many authors working on single crystals have only studied the first few percent of the deformation to avoid barrelling. Barrelling is inherent to single crystal deformation and the study of the steady state forces us to deal with it. The most awkward point is the definition of the stress: when the deformation is homogeneous, the stress is defined as the ratio of the applied force to the cross-section, but when it is heterogeneous, as in the CX samples, the cross-section is not constant along the sample.

Using the finite element method, Birch *et al.* [6] have determined the stress distribution developed in polycrystals during compression creep. Although studying single crystals, we can compare our strain distributions with their stress distributions. In the C samples, the iso-strain lines have a shape very similar to the iso-stress lines of the polycrystalline samples. A study such as that of Birch *et al.* could be done for our samples providing that one takes the anisotropy of the single crystals into account and that one can make a mathematical model for the material behaviour.

In order to improve the homogeneity of the deformation we have cut cubic samples. The improvement is hardly perceptible for the CX samples. The friction at the compression faces may prevent them from broadening enough to accomodate an homogeneous deformation; but until now we have not been able to lubricate the compression faces to confirm this because of the high chemical reactivity of Cu_2O at high temperature. It is also possible that barrelling and heterogeneous deformation would still exist even with lubrication because of the anisotropy of the glide systems.

Another problem which remains is to relate the macroscopic deformation to the glide elements. It has been observed that during high temperature deformation of C type parallelepipedic specimens only two glide planes operate out of four [1, 2, 7, 8] when for a cubic specimen the four glide planes operate [1]. No explanation has been put forward for this phenomenon. Probably the mechanical stability and the dislocation behaviour controls these macroscopic features of the deformation.

6. Conclusion

The compression test does not induce a simple stress state in the sample, particularly when the deformation is heterogeneous. When possible, the tension test must be preferred. When the compression test cannot be avoided, the study of the strain distribution allows the estimation of the heterogeneity of deformation: moreover it permits precise connection of the substructure observations to the true local strains. Finally, deformation produced by two active glide planes is much less homogeneous than deformation by four active planes.

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