Effects of Mechanical Constraint on the Strength of Ni₃Ge Single Crystals

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Dislocation structures in Ni₃Ge single crystals have been studied after deformation by [001] compression. In these tests octahedral slips govern the plastic deformation in absence of resolved stress for cube slips, causing anomalous strengthening throughout the temperature range studied, 77~500 K. Restriction of the operative shear systems by application of mechanical constraint is shown to increase the work hardening rate for the [001] compression. This phenomenon is opposite to the decrease of work hardening rate by the imposed constraint found for pure Cu in similar experiments. Three-dimensional dislocation structure has been examined by a high voltage electron microscope. Based on these observations, origin of the rapid work hardening and the anomalous strengthening in Ni₃Ge is attributed to a fine and non-planar dislocation structure developed in absence of resolved shear stress for the cube slips.

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1. Introduction

The anomalous strengthening in the L1₂ ordered alloys has been attributed to various types of dislocation structures and mechanisms of superdislocation motion involving cube cross slip below a peak temperature (T_p). Among them the most essential and common feature may be the non-planar structure of the superdislocations and its effect on dislocation motion,¹⁻⁹⁾ including the elastic anisotropy effect.^{10,11)} The dislocation locking causes rapid work hardening by exhaustion of mobile dislocations.¹²⁻¹⁶⁾ More strictly, decrease in the length of mobile dislocation segments causes the rapid work hardening in association with rapid increase in the K-W locking.¹⁸⁻²¹⁾

At this point, it may be important to recall that the anomalous strengthening, which is insensitive to the strain rate, would not be explained satisfactorily by considering the individual dislocation motion alone. In another words, mobility of the individual dislocation does not directly determine the strengthening of the athermal nature but the dislocation structure, resulting from the immobilization of the superdislocations, plays important role. The anomalous strengthening in both Ni₃Al and Ni₃Ge is controlled by the primary dislocations intersecting with the same primary dislocations cross slipped onto the cube planes in the $[\bar{1}23]$ compression, especially near the peak temperature (T_p).^{20,21)} Importance of the large cube cross slip in the anomalous strengthening has already been pointed out in several earlier studies.^{12,15)} In the [001] compression the above argument may not apply in absence of resolved shear stress for the cube slips. The situation must be different from the $[\bar{1}23]$ compression.

It is one of the aims in this study to clarify if the cross slip of superdislocations on to a cube plane plays a decisive role in determining the anomalous strengthening in the [001] compression. The yielding and work hardening behaviors will be examined at various temperatures under mechanical constraint imposed to restrict operative shear systems in the [001]

compression. The constrained and unconstrained experiments will also be made using pure Cu single crystals for comparison.

2. Experimental

Single crystals were grown from a stoichiometric Ni₃Ge compound by the usual Bridgman method in a crucible of 20 mm in diameter and 200 mm in length in an Ar atmosphere. The as-grown single crystals were vacuum-sealed in a silica tube and homogenized at 1473 K for 5 days. After the homogenization, chemical analyses revealed that the single crystals are composed typically of 75.8 at%Ni and 24.2 at%Ge. The specimens used for compression tests were cut from the rod to have a rectangular shape of $2.5 \times 2.5 \times 6.0$ mm³. The compression axis (C. A.) and the constraining faces were chosen along the [001] and (010) as shown in Figs. 1(a) and (b). The shear systems to be suppressed by the constraint are shown by filled marks in (a) and the two examples are shown by $\times$ marks in the schematic drawing of L1₂ structure in (b). After cutting and polishing, every specimen was sealed one by one in a silica tube, annealed at 1273 K for 2 h, cooled in the furnace and finally polished electrolytically by the same electrolyte used for TEM specimen preparation.

Compressive tests were made at 77 K in liquid nitrogen, at 300 K in air and at 500 K in silicone oil. The constraint was imposed on the (010) surfaces by holding the side surfaces with spacer blocks having desired thickness as shown in Fig. 2. Fine adjustment was needed for the spacer in order to make the effect of constraint observable at different temperatures and hence at different stress levels as shown in Fig. 2. The similar compression tests were made for pure Cu single crystals of the same orientation.

Thin foils for HVEM observations were prepared from the deformed specimen by slicing it along the (111) primary slip plane, namely the ones operated most predominantly. They were thinned down to 10~15 μ m at the thinnest portion by electrolytic jet polishing at 300 K, using an electrolyte,

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Fig. 1 The operative and non-operative shear systems in the [001] compression under the (010) plane constraint. (a) Slip system geometry shown in a stereographic projection. (b) Slip directions in the L_{12} lattice.

Fig. 2 Schematic drawing of the specimen set up for the [001] compression under the (010) plane constraint.

perchloric acid : ethanol : 2-butoxyethanol = 1 : 1 : 9.

The subsequent final polishing was made at 223 K in a different electrolyte,

perchloric acid : ethanol = 1 : 10.

The final polishing was interrupted several times at short intervals in order to check the foil thickness and to stop the polishing just before perforation. The final foil thickness was ap-

proximately $1\ \mu\text{m}$ at the thinnest center portion. The tedious and time consuming final polishing enabled us to observe the dislocations without rearrangement as already reported in the previous studies.¹⁷⁻²² Dimiduk *et al.*²³ have also pointed out the importance of avoiding thin-foil artifacts, especially in L_{12} intermetallic compounds in which superdislocations can dissociate into various types of partial dislocations.

The observations were made by a Hitachi-1250S HVEM at the accelerating voltage of 1000 kV with a double tilt specimen holder. Stereoscopic observations were made by tilting a specimen around the operating g vector by $8 \sim 10^\circ$ in the magnification range of $\times 8,000 \sim \times 15,000$.

3. Experimental Result

3.1 Stress strain behaviors

Representative stress strain curves obtained at three temperatures are compared in Fig. 3. In the figure, the circles marked on the stress-strain curves indicate the strains at which electron microscopic observations were made after unloading. Typical feature to be noted in the stress-strain curves is the rapid increase in the yield stress up to 500 K for the [001] compression in agreement with the earlier reports.^{24,25} Figure 4 shows the stress strain curves obtained at 300 K for pure Cu. According to the slip trace analyses, octahedral slips were confirmed for all the [001] compression tests. As expected, number of the operated shear systems was less in the constrained cases in both Ni_3Ge and pure Cu.

Interestingly, the work hardening rate increased notably by the imposed constraint while the yield stress did not change in significant magnitude in Ni_3Ge . The above observation in Fig. 3 is just opposite to the case of pure Cu single crystals shown in Fig. 4 where the work hardening rate is larger for the free [001] compression. The latter case appears to be the normal hardening due to dislocation-dislocation interaction in multiple slips. Another feature noted on the stress-strain curves in Fig. 3 is that the work hardening rate at a small strain is somewhat larger under the constraint. Such trend

Fig. 3 Representative stress strain curves obtained for Ni_3Ge at three temperatures by [010] compression with and without constraint. $\circ$ marks indicate the strains at which electron microscopic observations were made on the specimens sliced along the most predominantly operated slip planes.

Fig. 4 Representative stress strain curves obtained for pure Cu at 300 K by [010] compression with and without the (010) plane constraint. Note that the effect of the constraint is opposite to that found in Fig. 3. The work hardening rate is larger when deformed under no constraint.

is pronounced at the intermediate temperature upon acceleration of the K-W locking as also shown in Fig. 3. The origin of the effects of the constraint on the stress strain behaviors in Ni₃Ge will be inquired later in more detail with reference to the HVEM observations.

3.2 Dislocation structures after deformation

3.2.1 Deformed at 77 K

Figures 5(a) and (b) show the representative examples of the dislocation structures introduced by [001] compression at 77 K with and without constraint, respectively. The thin foil specimens were prepared by slicing the deformed crystals along the most predominantly operated (111) slip planes. The typical feature is that all the dislocations lying on the (111) planes have not cross slipped on to the (010) planes in visual magnitude neither in (a) nor in (b) as was confirmed by stereoscopic microscopy in these figures. Also, there is no clear difference in the dislocation density in agreement with equality of the final stress levels and strains found in Fig. 3.

The dislocation configurations are quite similar in (a) and (b) with screw rich character but not exactly aligned along the Burgers vector direction, being also similar to the $[\bar{1}23]$ compression of Ni₃Al at 300 K.²⁰⁾ This implies that the strengthening is affected to some extent by the K-W locking at this low temperature and even in absence of resolved shear stress for cube slips. Accordingly, the effect of the constraint on the stress strain curve, although it is small, is opposite in its nature to that observed for pure Cu in Fig. 4.

3.2.2 Deformed at 300 K

After compression by 3.9% along the [001] axis at 300 K, the dislocation configuration was altered significantly by application of the constraint. Burgers vectors of the operated shear systems were determined by examining the visibility as shown in Table 1 and in Fig. 6. In general, shape of dislocation became jerky after deformation under the constraint. Burgers vectors of the dislocations expected to operate under constraint are $[\bar{1}01]$ and $[101]$ as shown in Fig. 1. For example, dislocation segments A were identified to have the Burgers vector $[\bar{1}01]$ from the visibility shown in Figs. 6(a), (b) and (c) and the $g \cdot b$ values listed in the first column of Table 1. The Burgers vectors identified in Fig. 6 were $[\bar{1}01]$, $[0\bar{1}1]$ and $[1\bar{1}0]$ as shown schematically in Fig. 6(d). The latter two dislocations are expected to be immobile under the

Fig. 5 Dislocation structures observed after deformation at 77 K. (a) Deformed by 33.5% under the constraint. BD (Beam direction)=[211] and $g = 1\bar{1}\bar{1}$. (b) Deformed by 32.9% under no constraint. BD = $[121]$ and $g = \bar{1}1\bar{1}$. Effect of the constraint is not apparent at 77 K where K-W locks are not predominant.

Table 1 Visibility conditions for superdislocations.

		visible		invisible
		g						Schmid factor*
	b	$\pm\bar{2}02$	$\pm02\bar{2}$	$\pm\bar{2}20$	$\pm11\bar{1}$	$\pm\bar{1}11$	$\pm1\bar{1}\bar{1}$	
slips on (111) plane	$[\bar{1}01]$	±4	±2	±2	±2	±2		0.41
	$[\bar{1}10]$	±2	±2	±4		±2	±2	0.05
	$[01\bar{1}]$	±2	±4	±2	±2		±2	-0.35
slips on other {111} planes	$[101]$		±2	±2			±2	±0.41
	$[110]$	±2	±2		±2			±0.35
	$[011]$	±2		±2		±2		±0.05

* See IV.1. for Schmid factor in [001] compression under (010) constraint.

constraint as shown in Fig. 1. However, in the beginning of deformation these dislocations might have moved due to the constraint imposed in the compression tests. The curved segment C is judged to be the one introduced by the following reaction,

$$[\bar{1}01] + [01\bar{1}] = [\bar{1}10] \quad (1)$$

Density of the dislocations with $b = [0\bar{1}1]$ and $b = [1\bar{1}0]$ is, however, much lower than that of $b = [\bar{1}01]$ as found in Fig. 6.

Three-dimensional image of the dislocation configuration is also important in describing their mobility and interaction.

Fig. 6 Dislocation structures observed after deformation at 300 K by 3.9% under constraint. From the visibility tests shown in (a)~(c) coupled with Table 1, the Burgers vectors were determined and shown schematically in (d). Typical feature is that all the Burger vectors lie on the sliced (111) planes and the dislocation shape is not smooth but jerky.

Fig. 7 Stereo pair of micrographs, showing the dislocation structure in the same specimen as that shown in Fig. 6. (a) $BD = [111]$ and $g = 202$. (b) $BD = [121]$ and $g = \bar{1}1\bar{1}$. Different dislocations with $b = [\bar{1}01]$ $[101]$ were observed as groups in different locations (a) and (b) within the same specimen. These dislocations belong to the types, for which glide motion is not hindered by the (010) plane constraint.

Figures 7(a) and (b) show the different areas in the same thin foil specimen. Stereographic projection of the specimen is given in Fig. 7(a). It is apparent that different types of dislocations were introduced as groups in the different locations as shown in (a) and (b). This observation indicates local and predominant multiplication of one type of the dislocations over others in different areas under the constraint. The dislocations with $b = [\bar{1}01]$ are curved out of the (111) slip planes in (a) and those with $b = [101]$ penetrating through the foil are also curved off from the (111) slip planes in (b). The geometrical analysis has indicated that these dislocations have cross-slipped onto other octahedral planes.

For comparison, a typical example of the dislocation structures introduced by [001] compression under no constraint is shown in Figs. 8 and 9. Dislocations are smooth and curved, being clearly different in shape from the zigzag shape found

Fig. 8 Dislocations introduced by compression to 3.39% at 300 K under no constraint. (a) $\text{BD} = [112]$ and $g = \bar{1}\bar{1}1$. (b) $\text{BD} = [211]$ and $g = \bar{1}\bar{1}1$. (c) $\text{BD} = [121]$ and $g = \bar{1}\bar{1}1$. From the visibility tests shown in (a)~(c) coupled with Table 1, the Burgers vectors were determined to be $[\bar{1}01]$ and $[011]$.

in Figs. 6 and 7. According to the visibility tests shown in Figs. 8(a), (b) and (c), Burgers vectors of the major dislocations were identified to be $b = [\bar{1}01]$ and $[110]$. The $[\bar{1}01]$ dislocations are visible in (a) and (b) but invisible in (c) while the $[110]$ dislocations are visible in (a) but invisible in (b) and (c). These dislocations lie on the octahedral slip planes and very few of them have cross-slipped on to other octahedral slip planes such as marked by A in Fig. 9. It is apparent from Figs. 6–9 that the effect of the constraint at a small strain is to make the octahedral cross slip more active and to alter the dislocations to zigzag shape as seen in Figs. 6 and 7.

When the amount of deformation was increased up to

Fig. 9 Stereo pair of micrographs, showing the three dimensional dislocation structure in the same specimen examined in Fig. 8. $\text{BD} \sim [111]$ and $g = 220$. Note that a dislocation such as marked by A lying on the (111) slip plane with $b = [\bar{1}01]$ is curved off and cross slipped gently on to the $(\bar{1}\bar{1}1)$ plane. The short dislocation segments penetrating through the foil with $b = [011]$ lie on the $(\bar{1}\bar{1}1)$ planes. Jerky dislocations are absent here in contrast to Fig. 6.

12.2% under constraint and 20.0% under no constraint, dislocation density increased notably, accompanying dislocation clusters as shown in Figs. 10(a) and (b). These dislocations do not lie on planar slip planes as it is found by stereo microscopy in both of the figures. The effect of the constraint is to chop off the dislocations in fine scale as seen in (a). Also, the dislocation density is higher in (a) than in (b), despite that the strain is smaller for (a). The observed refinement in the dislocation structure is in good accordance with the larger work hardening found under the constraint in Fig. 3. Origin of this structured refinement will be discussed in 4.2.

Reduction in number of the slip systems, thus, causes extra hardening in the $[001]$ compression, implying that usual dislocation-dislocation cutting is not essential in the work hardening when plastic deformation proceeds by superdislocation motion. Such dislocation behavior is quite different from the usual work hardening found in pure Cu, in which work hardening was smaller under constraint as shown in Fig. 4. Thus, the extra hardening observed under constraint in Ni_3Ge must be attributed to the characteristic behavior of the dislocation motion involving K-W locks.

3.2.3 Deformed at 500 K

The anomalous temperature dependence was extended to higher temperature region in the $[001]$ compression in absence of the resolved shear stress for cube slips as shown in Fig. 3. The constraint reduced the activity of particular operative slip systems as shown below in the visibility tests of Figs. 11(a), (b) and (c). Dislocations introduced in these tests were mostly of $[\bar{1}01]$ and $[101]$ types whose motion is not restricted by the constraint. Screw components were more predominant after deformation at 500 K than at 77 and 300 K. This fact again implies the important role of the K-W locks at a high temperature even in absence of resolved shear stress

Fig. 10 Stereo pair of micrographs, showing the dislocation structure after deformation at 300 K by 12.2% under constraint (a) and by 20.0% under no constraint (b). $BD \sim [12\bar{1}]$ and $g = 111$ for both of the micrographs. Note that high density of curled dislocations are distributed three dimensionally and tangled in fine scale in both cases. Dislocation density is, however, higher and more localized in (a) than in (b), despite that the amount of deformation was smaller for (a).

for the cube slips. Accordingly, effect of the constraint was masked by the predominance of the K-W locking and was not distinct at 500 K as shown in Fig. 3.

Concurrently, the work hardening was smaller at 500 K than at 300 K in contrast to the notable increase in the yield stress. From the stereo micrographs shown in Figs. 12(a) and (b), it is found that dislocation structure changes with strain in a quite similar manner at 500 and 300 K. Thus, the flow stress at a large strain is nearly the same at these two temperatures as shown in Fig. 3. However, the smaller yield strength at the lower temperature brings about the larger work hardening at 300 K where the effect of constraint appears most predominantly.

Fig. 11 Dislocation structures observed after deformation at 500 K by 4.8% under constraint. Thin foil specimen was sliced along the (101) plane. From the visibility tests shown in (a)~(c) coupled with Table 1, the Burgers vectors were determined. Typical feature is that all the dislocations are straighter than those introduced at lower temperatures, indicating difficulty of cross slip onto other octahedral slip planes due to pronounced K-W locking.

4. Discussion

4.1 Stress state under the (010) constraint

The usual Schmid law can not be applied when a specimen is loaded under constraint. In the present case, the constraint imposed on the (010) plane may be expressed by

$$\varepsilon_{22} = 0 \quad (2)$$

$$\sigma_{33} = \sigma_A, \quad \sigma_{11} = 0 \quad (3)$$

Fig. 12 Stereo pair of micrographs, showing the dislocation structure after deformation at 500 K under donstraint. BD $\sim [101]$ and $g = 20\bar{2}$ for (a) after deformation by 4.8%. BD $\sim [121]$ and $g = \bar{1}\bar{1}1$ for (b) after deformation by 13.4%. Note that introduction of curled dislocations in fine scale in (b) differs from the rather straight dislocations found at a smaller strain in (a).

where σ_A is the unidirectional stress applied along the $[001]$ axis. The Fooke's law to relate σ_{ij} and ε_{kl} is written by Mura²⁶⁾

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl} \quad (4)$$

where C_{ijkl} for a cubic lattice is given by

$$C_{ijkl} = \lambda\delta_{ij}\delta_{kl} + \mu\delta_{il}\delta_{jk} + \mu\delta_{ik}\delta_{jl} + \mu'\delta_{ijkl} \quad (5)$$

Here, all $\delta_{ijkl} = 0$ except $\delta_{1111} = \delta_{2222} = \delta_{3333} = 1$ and

$$\lambda = C_{1122}, \quad \mu = C_{1212}, \quad \mu' = C_{1111} - C_{1122} - 2C_{1212}$$

From equations (2), (3), (4) and (5) we obtain

$$\sigma_{22} = [\lambda/(2\lambda + 2\mu + \mu')]\sigma_A \quad (6)$$

By taking the x' , y' and z' axes along the $[011]$, $[11\bar{1}]$ and $[\bar{2}1\bar{1}]$ directions, the resolved shear stress $\sigma_{x'y'}$ for one of the

slip systems $[011](111)$ not operative under the constraint is now given by

$$\sigma_{x'y'} = [(\lambda + 2\mu + \mu')/(2\lambda + 2\mu + \mu')/\sqrt{6}]\sigma_A \quad (7)$$

Thus, Schmid factor is reduced by the constraint. More importantly, $\sigma_{x'y'}$ must be reduced significantly by operation of the shear system itself since the stress of opposite sign is generated by suppression of the crystal rotation under the constraint.

On the other hand, resolved shear stress for the unconstrained slip system $[10\bar{1}](111)$ is not altered by the presence of σ_{22} , since the $[10\bar{1}]$ direction lies on the (010) constraint plane. Thus, Schmid factor for the $[10\bar{1}](111)$ slip system is given by $1/\sqrt{6}$ in the $[001]$ compression regardless of the presence of the constraint. The yield stress is, thus, not affected by the (010) constraint as found in Figs. 3 and 4.

Interestingly, the work hardening rate increased notably upon reduction in number of the slip systems under the constraint as shown in Fig. 3, especially at the intermediate temperature 300 K. It suggests that the imposed constraint increases the work hardening rate of the Ni₃Ge not by mutual cutting of dislocations but by the structural change of the dislocations in the individual slip systems. On the contrary, the mechanical constraint decreased the work hardening rate in pure Cu as shown in Fig. 4. The latter case is the usual work hardening caused by dislocation cutting in the multiple slip orientation.

4.2 Acceleration of work hardening by constraint

At all the three temperatures examined, the work hardening rate was increased by suppression of number of the operative slip systems as shown in Fig. 3. This observation is opposite to the usual expectation that the work hardening rate must decrease with decrease in number of the operative shear systems if the dislocation motion is hindered by other dislocations. An example of usual hardening by multiple slips is shown in Fig. 4. The unusual behavior of the work hardening in the anomalous strengthening region may be attributed to the $\{111\}$ type of cross slips coupled with K-W locks in absence of resolved shear stress for the cube slips in the $[001]$ compression. In addition to this, cross slip on to octahedral planes must be enhanced more in the ordered Ni₃Ge than in pure fcc metals since the superdislocations tend to take the screw orientation by K-W locking. The constraint imposed by the axis rotation must also be taken into account in the compression tests.

At this point, it might be helpful to remind the stereographic projection and the slip systems shown in Fig. 1. Only two types of the shear directions are operative under the constraint but each of them lie on two $\{111\}$ planes and hence on all the four $\{111\}$ planes in total. Moreover, the two types of the shear systems were located in different areas as shown in Fig. 7. Then, it is easily imagined that dislocation reaction will take place between those having the same Burgers vector, contributing to the extra hardening. This type of hardening is quite similar to the rapid work hardening caused by the (010) cross slip in the $[123]$ compression.²⁰⁾ The dislocation reaction between those with the opposite signs of the same Burgers vector has also been described in the textbook

by Read.²⁷⁾

In support of this expectation, many of the dislocation segments were curved and were not lying on planner slip planes as shown in Figs. 10, 11 and 12. The work hardening rate will, thus, increase by cross slip of the screw dislocations on to octahedral planes and by mutual trapping of the dislocations with the same Burgers vector but lying on different slip planes. The other factor that might contribute to the excess hardening is simply the increase in the shear strain in each slip system by reduction in number of the operative shear systems under the constraint. The latter factor must, of course, be important when the work hardening behavior is not seriously affected by operation of other slip systems as in the present case.

4.3 Optimum condition for extra hardening by constraint

Amount of the excess hardening was largest at 300 K in the present tests. This fact may be explained by considering relative contribution of the K-W locking and the dislocation-dislocation interaction to the hardening. The octahedral cross slip will occur more frequently at a higher temperature when a superdislocation takes screw configuration but not capable of making cross slip onto the cube planes in absence of a resolved shear stress. The excess hardening due to the dislocation cutting of the type mentioned above will increase when K-W locking induces screw components more frequently at a higher temperature. However, as K-W locking becomes more stronger at the higher temperature, as it is found in the straight screw dislocations in Fig. 11, increase in the anomalous hardening by the K-W locking will bias the effect of constraint as shown in Fig. 3. Thus, there is an optimum condition in producing the maximum effect of the constraint which is attained at the intermediate temperature as observed in the stress strain curves at 300 K in Fig. 3.

5. Conclusions

Single crystals of Ni₃Ge and pure Cu oriented for [001] compression have been deformed with and without mechanical constraint on the (010) surfaces in the temperature range 77~500 K. Thin foil specimens were prepared to have the foil surfaces along the most predominantly operated slip planes and examined by HVEM at the accelerating voltage of 1000 kV. From the three dimensional analysis of the dislocation structure the following conclusions were obtained.

(1) Work hardening rate in Ni₃Ge increased notably by the (010) constraint, especially at 300 K, while the yield stress did not change by the constraint.

(2) Dislocations were curved on to other octahedral planes to have three- dimensional structure under the con-

straint, most markedly at 300 K in accordance with the extra work hardening.

(3) Dislocation density increased significantly by the constraint upon reduction in the operative shear systems and by mutual locking of the dislocations of the same Burgers vector but lying over different slip planes.

(4) In contrast to Ni₃Ge, work hardening rate in pure Cu decreased under the imposed constraint due to reduction in number of the operative shear systems.

(5) The present observations confirm that development of the three dimensional dislocation structure plays an important role in the rapid work hardening which is essentially unaffected by interaction with other shear systems in the ordered Ni₃Ge.

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