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Effect of orientation on work softening in aluminum single crystals

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A single crystal of aluminum, prestrained at low temperature, exhibits a yield drop when it is deformed at a sufficiently higher temperature and this phenomenon has been termed the work softening. In this study, three types of aluminum single crystals with various tensile orientations were stretched at 77 K and 293 K, to clarify the effect of orientation on this work softening phenomenon. The single crystal orientated for single slip shows work softening when deformed at 293 K after prestraining satisfactorily at 77 K. In the deformation of this crystal at 293 K, a coarse slip accompanied by an intimate cross slip, was observed. The single crystal orientated along ⟨1 0 0⟩ also shows work softening at 293 K after giving moderate elongation at 77 K and a clustered slip accompanied by a prominent cross slip was observed in the deformation at 293 K. However, in the deformation of the single crystal orientated along ⟨1 1 1⟩ at 293 K, only a fine triple slip was observed and work softening was not observed even when prestrained to a large strain at 77 K. It is thought that the work softening found at 293 K after prestrain at 77 K is associated with the occurrence and propagation of coarse slip accompanied by cross slip.

1. Introduction

Stokes and Cottrell [1, 2] pointed out that a drop of yield stress was observed with a decrease of flow stress when an aluminum single crystal was deformed at a sufficiently higher temperature after elongation of several per cent at low temperature. The yield drop is generally associated with the interaction between dislocations and impurities or vacancies. However, they demonstrated that this phenomenon did not originate from the common mechanism of the yield drop, and that it could be explained as a result of work softening.

Work softening of the same kind has also been observed in aluminum single crystals and magnesium single crystals when deformed at a low strain rate after impact deformation [3–5]. In both above cases, it is thought that this work softening is caused by the collapse of the metastable structure of dislocations formed at the first deformation, or by the cross slip avoiding obstacles formed at the first deformation.

It is well known that the dislocation structures formed during work hardening or the ease of cross-slip are greatly influenced by the crystal orientation. Therefore, it is supposed that the work softening may be greatly influenced by the crystal orientation.

However, this effect has been studied scarcely at all, save for the work of Kashihara *et al.* [6], using aluminum single crystals deformed at high strain rate.

In the present study, three types of aluminum single crystals, a crystal with ⟨1 0 0⟩ tensile orientation (for which cross slip can most easily occur), a crystal with ⟨1 1 1⟩ tensile orientation (for which the occurrence of cross slip is most difficult) and a common single crystal for single glide, are prepared. In order to clarify the effect

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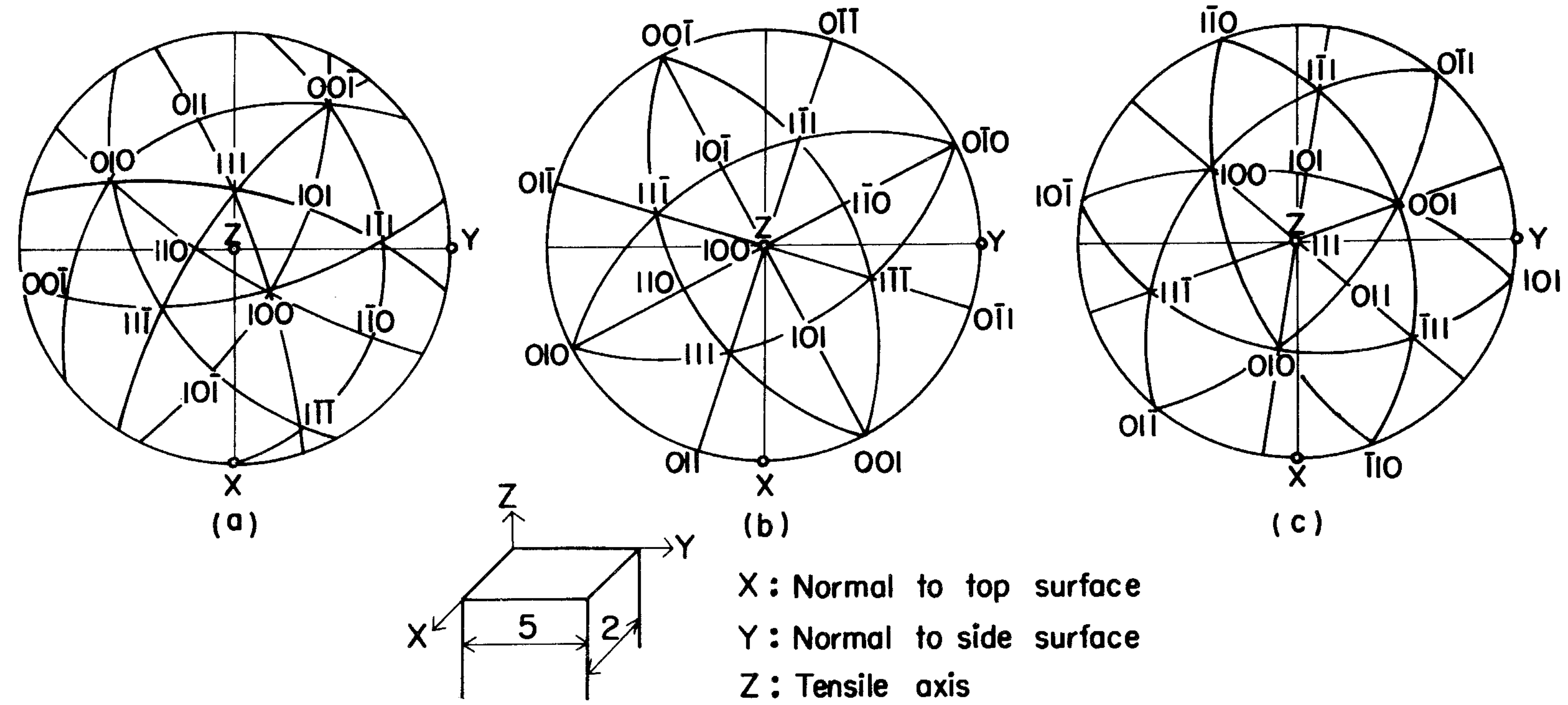

Figure 1 Orientation of single crystals: (a) single-slip orientated single crystal; (b) ⟨1 0 0⟩ orientated single crystal; (c) ⟨1 1 1⟩ orientated single crystal.

of orientation on work softening and to discuss the relation between the cross slip and work softening, these single crystals are prestrained at 77 K and continuously strained at room temperature.

2. Experimental procedure

The material used in this experiment was 99.99% pure aluminum. Single crystal plates, having an orientation controlled by seeding, were grown by the Bridgman method in a vacuum. Specimens with a gauge dimension of 2 mm x 5 mm x 15 mm were cut from the single crystal plates by spark cutting and then annealed at 823 K for 3 h. All specimens were polished mechanically and electrolytically before a tensile test.

The orientation of these single crystal specimens are shown in Fig. 1a, b and c. One of these single crystals is deformed by single slip at the early stage of plastic deformation (Fig. 1a). The others have an ⟨1 0 0⟩ and an ⟨1 1 1⟩ tensile orientation, the former deformed by quadruple slip, the latter by triple slip (Fig. 1b and c).

In this paper, the single crystals shown in Fig. 1a, b and c are termed single-slip-orientated single crystals, ⟨1 0 0⟩ orientated single crystals and ⟨1 1 1⟩ orientated single crystals, respectively. The accuracy of orientation of the single crystals could be controlled to within ± 1°. Tensile tests were carried out with a strain rate of 4×10^{-5} $\sec^{-1}$ at 77 K (in liquid nitrogen) and at 293 ± 1 K. Slip line observation was made at each stage of deformation.

3. Results and discussion

3.1. Work softening and slip line observation

3.1.1. Single-slip-orientated single crystal

The stress–strain curve of the single-slip-orientated single crystals, stretched by 5.5% strain at 77 K and continuously at 293 K, is shown in Fig. 2a. The flow stress increased rapidly after showing a short Stage I at 77 K, but scarcely increased as deformation proceeds after the yield drop of 0.75 MN m^{-2} (about 3% of whole flow stress) at 293 K.

This yield drop is similar to that reported as work softening by Stokes and Cottrell [1]. They demonstrated that this yield drop is not due to the ageing treatment, since the purity of material used in their experiment is high enough, and concluded that it is not a yield point drop caused by strain ageing.

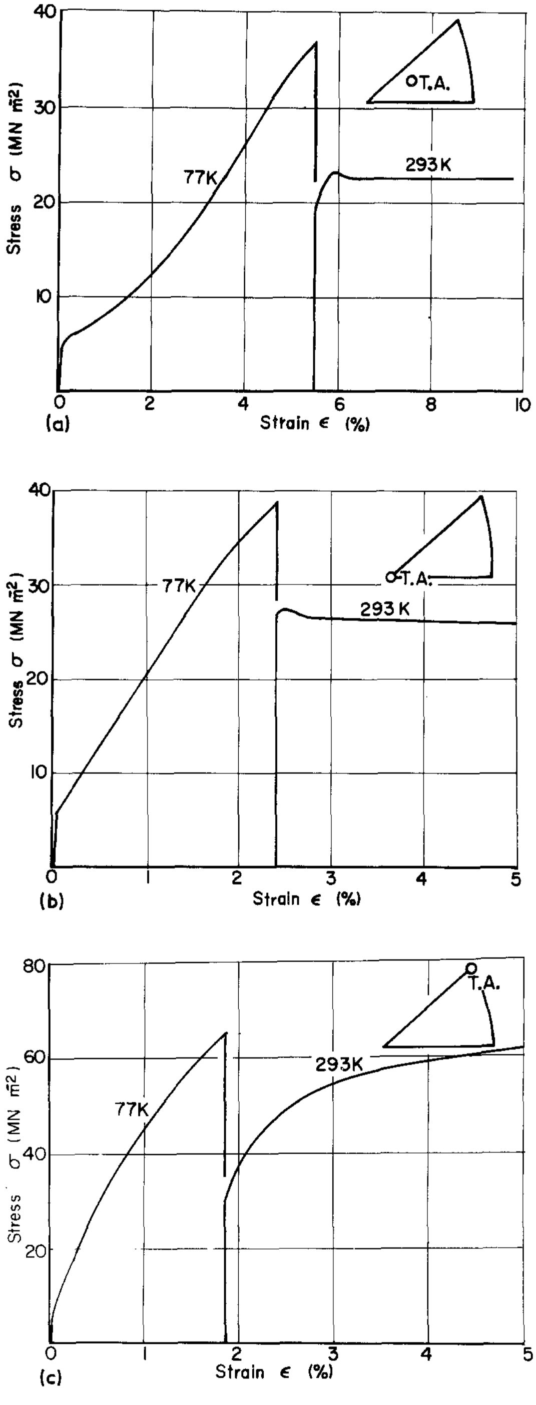

Figure 2 Stress–strain curves for a single crystal at 77 K and 293 K: (a) single-slip-orientated single crystal; (b) ⟨1 0 0⟩ orientated single crystal; (c) ⟨1 1 1⟩ orientated single crystal.

Yield drop has also been observed in single crystals of nickel, aluminum [7] and copper [8] when again loaded at the same temperature after unloading. This is well known as the Haasen–Kelly

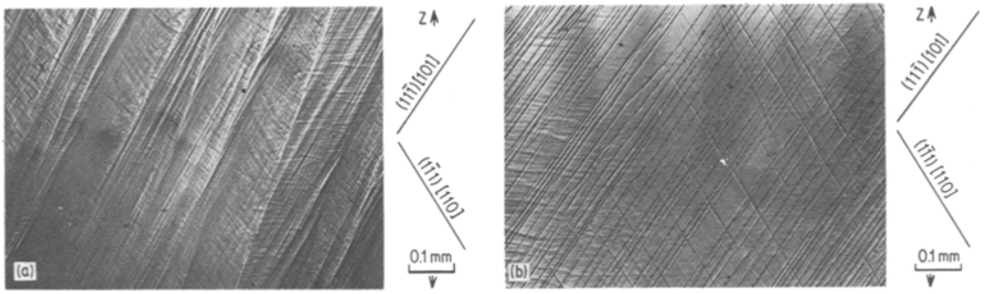

Figure 3 Slip lines observed in single-slip-orientated single crystal: (a) specimen electrolytically polished at zero strain, slip lines observed at 77 K at a strain of 5.5%; (b) specimen electrolytically polished after elongation of 5.5%, slip lines observed at 293 K at a strain of 6.8%.

effect [7]. But, in this case, the magnitude of yield drop is less than 1% of the whole flow stress and the stress–strain curve instantly coincides with the extention of the prior stress–strain curve. Therefore, it is evident that the yield drop observed in our experiment is not the Haasen–Kelly effect.

Fig. 3a shows the top surface of a single-slip-orientated single crystal which was stretched by 5.5% strain at 77 K. Fig. 3b shows the top surface of the same specimen continuously stretched to 6.8% strain at 293 K subsequent to 5.5% prestrain at 77 K. In this study, for the convenience of observation, the specimen surface was electrolytically polished several times during the experiment. It was found that the primary slip: $(1\,1\,\bar{1})\ [1\,0\,1]$, and the conjugate slip: $(1\,\bar{1}\,1)\ [1\,1\,0]$, occurred strongly in deformation at 77 K, while in the deformation at 293 K, the coarse primary slip was observed immediately and the coarse conjugate slip was also observed. These slips are clearly different from those observed at deformation at 77 K and were accompanied by an intimate cross slip.

The macroscopic observations of slip lines on a single-slip-orientated single crystals deformed at 293 K are shown in Fig. 4. In the deformation at 293 K, the coarse slip began to appear at either end of the specimen and propagated to the central region and to the other end of the specimen. Therefore, the flow stress of the single-slip-orientated single crystal is not increased in the deformation at 293 K in a similar way to the propagation process of the Lüders band.

3.1.2. ⟨1 0 0⟩ orientated single crystal

When the ⟨1 0 0⟩ orientated single crystal was stretched at 293 K after a prestrain of 2.4% at 77 K and unloaded, its flow stress remained almost constant after the yield drop of $1.1\ \mathrm{MN\,m^{-2}}$ (about 4% of the whole flow stress) (see Fig. 2b).

In the deformation of this single crystal at 77 K, only the fine quadruple slip operated (see Fig. 5a) and so its flow stress increased rapidly, as reported previously by the present authors [9]. On the other hand, in order to avoid the obstacles formed in the deformation at 77 K, the clustered slip, accompanied by the prominent cross slip characteristic of the ⟨1 0 0⟩ orientated single crystal [10], was found in the deformation at 293 K (see Fig. 5b). As the deformation proceeded at 293 K, this clustered slip propagated from both ends of the specimen towards the central part (see Fig. 6), in a similar way to the propagation of the Lüders band, making the flow stress constant similar to that of the single-slip-orientated single crystal deformed at 293 K after prestrain at 77 K.

3.1.3. ⟨1 1 1⟩ orientated single crystal

Fig. 2c is the stress–strain curve of the ⟨1 1 1⟩ orientated single crystal, which was unloaded and stretched continuously at 293 K after being elongated by 1.8% prestrain at 77 K. Its flow stress increased rapidly due to the triple slip, showing the characteristic of ⟨1 1 1⟩ orientated single crystal deformed at 77 K. However, the stress–strain curve of this single crystal at 293 K is totally different from that of the single-slip-orientated single crystal or the ⟨1 0 0⟩ orientated single crystal; the flow stress of the ⟨1 1 1⟩ orientated single crystal increased as the deformation proceeded at 293 K without showing yield drop. Only the triple slip was observed in the ⟨1 1 1⟩ orientated single crystal deformed at 77 K (see Fig. 7a). The slip found in this crystal deformed at 293 K is neither the coarse slip, as was observed in the single-slip-orientated single crystal, nor the ⟨1 0 0⟩ orientated single crystal deformed at 293 K, but was the triple slip (see Fig. 7b).

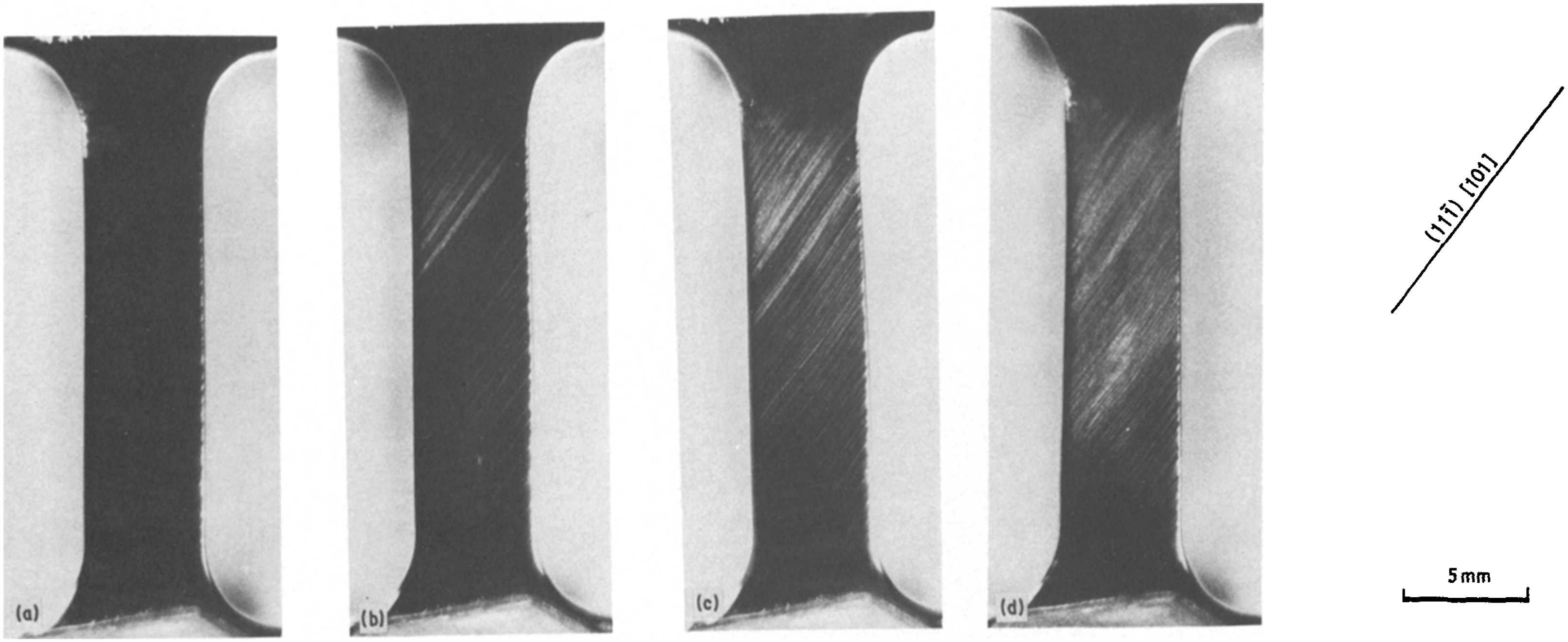

Figure 4 Propagation of coarse slip lines accompanied by intimate cross slip in a single-slip-orientated single crystal. Specimen electrolytically polished after elongation of 5.5% strain at 77 K. Slip lines observed: (a) at 293 K at a strain of 5.7%; (b) at 293 K at a strain of 5.9%; (c) at 293 K at a strain of 6.8%; (d) at 293 K at a strain of 9.7%.

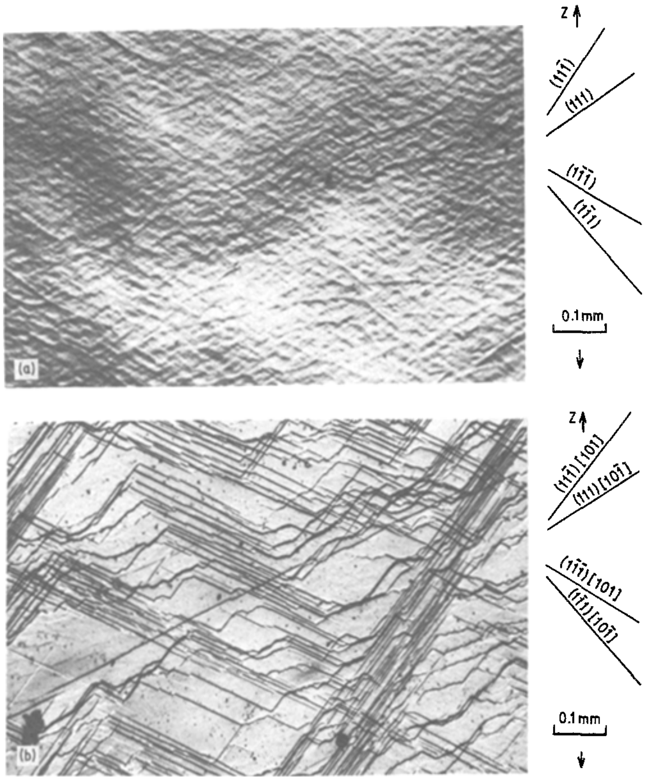

Figure 5 Slip lines observed in ⟨100⟩ orientated single crystal: (a) specimen electrolytically polished at zero strain, slip lines observed at 77 K at a strain of 2.4% (fine multiple slip); (b) specimen electrolytically polished after an elongation of 2.4%, slip lines observed at 293 K at a strain of 7.3% (coarse slip accompanied by prominent cross slip).

These results suggest that the work softening observed was closely related to the activation and the propagation of coarse slip.

3.2. Alternate deformations and work softening

The stress–strain curves of the single-slip-orientated single crystal, the ⟨1 0 0⟩ orientated single crystal and the ⟨1 1 1⟩ orientated single crystal, deformed alternately at 77 K and 293 K, are shown in Fig. 8a, b and c.

In the deformation of single-slip-orientated single crystals, work softening began after being prestrained by 3.4% strain and was clearly seen after 5.3% prestrain. This means that the work softening was caused after there was sufficient formation of obstacles to slip, such as the sessile dislocations due to multiple slip.

The ⟨1 0 0⟩ orientated single crystal clearly showed work softening after being prestrained by 2.7%. It is thought that this ease of work softening in the ⟨1 0 0⟩ orientated single crystal corresponded to the easier formation of obstacles to slip in this single crystal; the single-slip-orientated single crystal was deformed by single slip at an early stage of deformation, but quadruple slip was activated immediately after yielding in this single crystal.

In the ⟨1 1 1⟩ orientated single crystal, work softening was not observed even after prestraining to a large strain, despite the active operation of the triple slip. It is though that work softening was involved with not only the activation of multiple slip but also the occurrence of cross slip.

The ratio of the flow stress at 77 K and 293 K at the same strain has been calculated from Fig. 8 and the relation of the ratio of the flow stresses and the strain is shown in Fig. 9. In Fig. 9a and b, the upper yield stress (before the work softening occurs) and the lower yield stress (the constant flow stress after work softening) are taken as the flow stress at 293 K. The curves of the single-slip-orientated single crystal, the ⟨1 0 0⟩ orientated single crystal and the ⟨1 1 1⟩ orientated single crystal are in very good agreement with each other in Fig. 9a. It is shown that the ratio of flow stress at 77 K and 293 K is sufficiently constant that, if the work softening is ignored, it is independent of the crystal orientation. However, in Fig. 9b, the three curves diverge as the deformation proceeds, and their departure is related to the magnitude of the work softening in the

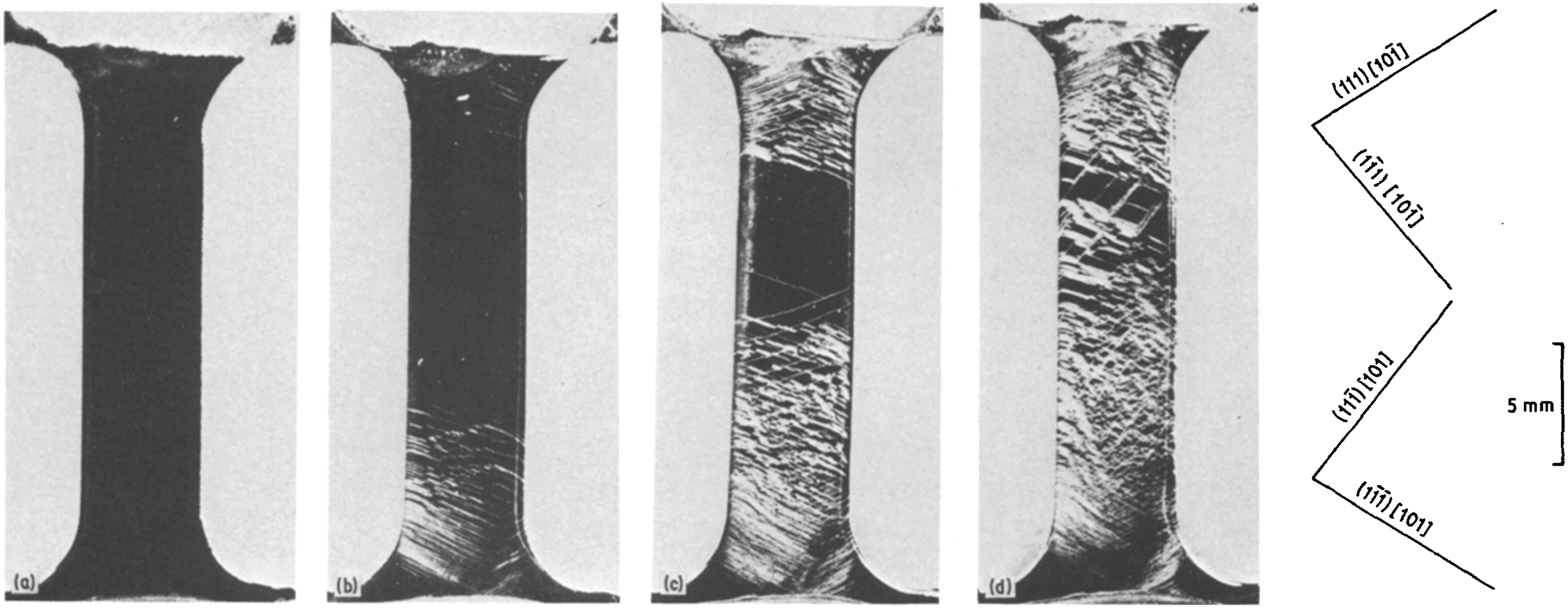

Figure 6 Macroscopic observation of slip lines in ⟨1 0 0⟩ orientated single crystal: (a) specimen electrolytically polished at zero strain, slip lines observed at 77 K at a strain of 2.4%; (b–d) specimens electrolytically polished at a strain of 2.4%, slip lines observed at 293 K at strains of 2.8%, 4.1% and 7.3%, respectively.

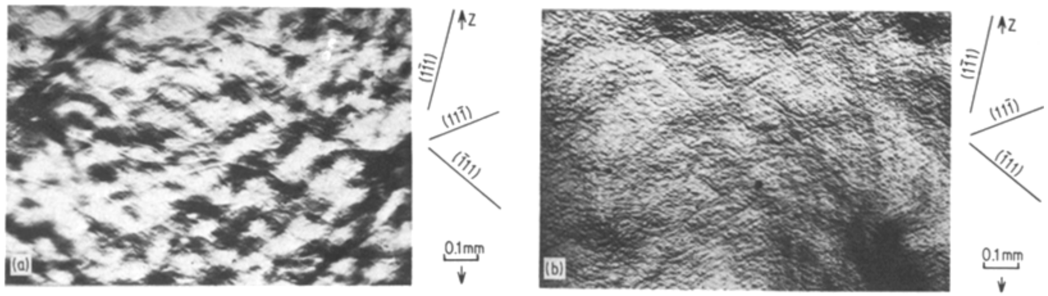

Figure 7 Fine multiple slip lines observed in ⟨1 1 1⟩ orientated single crystal: (a) specimen electrolytically polished at zero strain, slip lines observed at 77 K at a strain of 1.8%; (b) specimen electrolytically polished at a strain of 1.8%, slip lines observed at 293 K at a strain of 5.8%.

respective single crystals. It is evident that the work softening was severely affected by the tensile orientation.

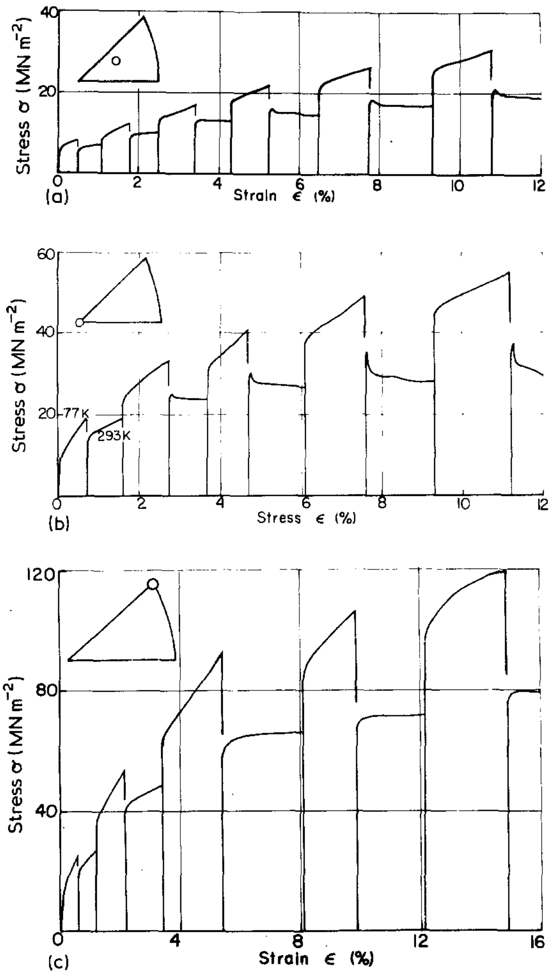

Figure 8 Stress–strain curves taken alternately at 77 K and 293 K. (a) Single-slip-orientated single crystal. (b) ⟨1 0 0⟩ orientated single crystal. (c) ⟨1 1 1⟩ orientated single crystal.

3.3. Work softening and tensile orientation

When work softening occurred, a coarse slip, accompanied by the intimate cross slip and by the prominent cross slip, was found in the single-slip-orientated single crystal and in the ⟨1 0 0⟩ orientated single crystal. However, no coarse slip was found, except the fine triple slip, in the ⟨1 1 1⟩ orientated single crystal, which did not show work softening. In consequence, it is thought that the occurrence of work softening is due to the activation of the coarse slip avoiding obstacles formed in the crystal during deformation at 77 K.

Miura *et al.* [11] studied the orientation dependence of the stress required to make cross slip which indicates the ease of cross slip, and they reported that it depends on the ratio M of the shear stress on the cross slip system τ_{cross} to that of the primary slip system τ_{prim}: $M = \tau_{\text{cross}}/\tau_{\text{prim}}$. The value of M is found to be equal to 1 for ⟨1 0 0⟩ orientation and equal to -1 for ⟨1 1 1⟩ orientation, if account is taken of the direction of resolved shear stress on the cross slip plane, and M is zero on the ⟨1 1 0⟩–⟨2 1 1⟩ line (see Fig. 11 of [11]). Therefore, the ⟨1 0 0⟩ orientation is the one where cross slip most easily occurs, and it is the most difficult for cross slip to occur for the ⟨1 1 1⟩ orientation.

In the present study, it has been pointed out that the presence of this work softening is dominated by the activation of the cross slip, and that the easier the cross slip, the larger the magnitude of work softening. As a result, the work softening observed at room temperature after deformation at 77 K was dependent on the crystal orientation.

4. Conclusion

The effect of crystal orientation on work softening in aluminum single crystals, found at room temperature after deformation at 77 K, was studied

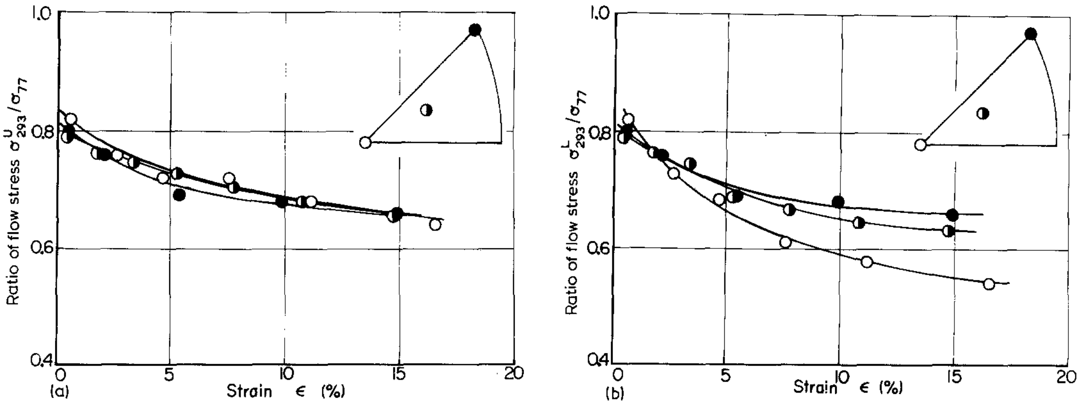

Figure 9 Ratio of flow stresses at 77 K and 293 K for three sorts of single crystals. Curves (a) and (b) were taken from the values of upper and lower yield stress, σ^U_{293}, σ^L_{293}, at 293 K, respectively.

using single-slip-orientated single crystals, ⟨1 0 0⟩ orientated single crystals and ⟨1 1 1⟩ orientated single crystals. The single-slip-orientated single crystals and the ⟨1 0 0⟩ orientated single crystals exhibited work softening, and the ⟨1 1 1⟩ orientated single crystal did not exhibit work softening at 293 K after moderate elongation at 77 K.

When work softening was observed, coarse slip accompanied by intimate cross slip and by prominent cross slip was found in the single-slip-orientated single crystals and in the ⟨1 0 0⟩ orientated single crystals, respectively, while only fine triple slip was observed in the ⟨1 1 1⟩ oriented single crystals at 293 K after the large deformation at 77 K.

It is thought that work softening found at 293 K for specimens after deformation at 77 K was caused by the occurrence and propagation of coarse slip accompanied by cross slip and that the orientation dependence can be explained by the ease of cross slip.

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