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SINGLE-CRYSTAL ALUMINIUM**

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**ELECTRON MICROSCOPIC OBSERVATION ON THE DISLOCATION CONFIGURATION CORRELATIVE WITH THE HIGH-TEMPERATURE INTERNAL-FRICTION PEAK IN 99.999 wt % SINGLE-CRYSTAL ALUMINIUM**

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**Abstract** - The dislocation structure in sheet specimens of 99.999 wt % aluminium single crystals was observed by transmission electron microscope (TEM) in order to explore the micro-mechanism corresponding to the appearance and disappearance of the internal-friction peak around 365°C ( $f = 1$  Hz). /1/ Internal-friction measurements were made with an inverted torsion pendulum on sheet specimens having a thickness of 1 mm or smaller and a width of 4.5 mm. The same specimen was thinned down after the internal-friction measurements by chemical polishing and a double jet electrolytic polishing to films about 700 nm thick for TEM observations. The results of internal-friction measurements were compared with TEM observations.

**I. Introduction** - An internal-friction peak situated at 365°C ( $f = 1$  Hz) was recently observed by KÈ et al. in 99.999 % aluminium single crystals. Subsequent experiments /2/ showed that this peak appears only when the single-crystal specimen was prepared by dynamic strain-annealing method and annealed subsequently at a temperature above 550°C. Furthermore, this peak was reduced or suppressed when the single-crystal specimen was deformed at room temperature or under creep conditions. Meanwhile, the internal friction background beyond the temperature of the 365°C peak was considerably increased. In order to correlate the change of dislocation configuration in the specimen with the appearance and disappearance of the 365°C peak, TEM observations were made on the same specimen after the internal friction measurements.

**II. Experimental Procedure** - An aluminium sheet (99.999 %) of  $120 \times 4.5 \times 1$  mm<sup>3</sup> was annealed at 500°C for 0.5 h and then stretched at room temperature to 2.3 % elongation. This sheet was put inside a tube furnace with a predetermined temperature gradient along the tube. The hottest part of the specimen was kept at a temperature of about 550°C. The sheet was displaced in the furnace with a uniform speed of 2.5 cm/h. The single crystal specimen thus prepared was mounted in an

inverted torsion pendulum for internal friction measurements. After the measurements, the specimen was cut to several portions and thinned down by chemical polishing to 0.1 mm by soaking in a solution of NaOH (20 g) and H<sub>2</sub>O (100 ml) at 70°C. It was then treated by a double jet electrolytic polishing to films about 700 nm for TEM observations in a high resolution electron microscope (TEM-200 cx) at a working voltage of 200 kV.

### III. Experimental Results -

III. 1. A single-crystal specimen (containing about 2--3 bamboo boundaries along a length of 120 mm) of 1 mm thick was mounted in an inverted torsion pendulum, and internal friction measurements were taken with ascending temperatures. The internal friction curve thus obtained is shown by curve 1 of Fig. 1. It is seen that the internal friction increases monotonously with temperature and a small plateau appeared around 410°C. This specimen was annealed at 600°C for 2 h and internal-friction measurements started at 550°C in descending temperatures with a speed of 2°C/min. An internal friction peak appeared pronouncedly at 410°C ( $f = 1.7$  Hz) with a height of 0.02 as shown by curve 2 of Fig. 1. It can be seen that this peak is the 365°C peak with  $f = 1$  Hz.

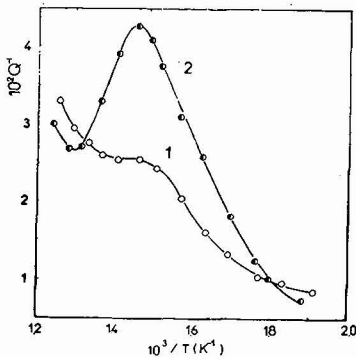


Fig. 1. Internal friction curves of 99.999 aluminium single crystal prepared by dynamic strain annealing method: Curve 1. Measured in ascending temperatures. Curve 2. After an annealing at 600°C for 2 h.  $f = 1.7$  Hz.

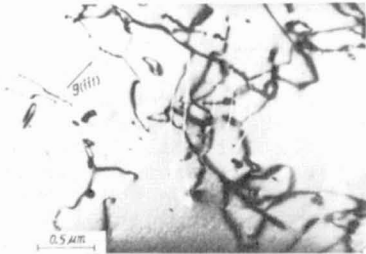


Fig. 2. Dislocation configuration in 99.999 aluminium single crystal after an annealing at 600°C for 2 h.

The dislocations in the specimen exhibiting a "365°C peak" observed by TEM in the film prepared from this specimen are dispersive (polygonization boundaries were not observed). They wove together to form a uniform spatial network with many nodes as shown in Fig. 2. Diffraction contrast analysis by tilting the film showed that the dislocations composing the spatial network are not situated on the same plane. Such a dislocation configuration is a stable structure formed by annealing at an elevated temperature above 550°C. The dislocations forming the network are of the edge-type. Repeated measurements showed that the dislocation density is about  $10^6$  to  $10^9$ /cm<sup>2</sup>. The dislocation segment between the spatial nodes is quite long with an average length of about 0.5--1 μm. Diffraction contrast analysis showed that screw dislocation does not exist.

III. 2. Another single-crystal specimen prepared by dynamic annealing method was mounted in the torsion pendulum apparatus and the internal friction curve measured with ascending temperatures is shown

by curve 1 of Fig. 3. The internal friction increases monotonously with temperature and a plateau appeared at a temperature region lower than that shown by curve 1 of Fig. 1. When the temperature is raised to 450°C and internal friction is taken with descending temperatures a hump appeared around the region of 350--450°C. This indicates that the 450°C annealing is not sufficient to have the 365°C peak well developed as in the case shown by curve 2 of Fig. 1 after an anneal of 600°C.

TEM observations showed that the dislocations in the specimen film wove into a complex irregular structure as shown in Fig. 4 (a). The average distance between the nodes is much shorter as compared with that shown in Fig. 2. There is only a small proportion showing the spatial network structure as in Fig. 4 (b). The dimension of the network is large and very irregular.

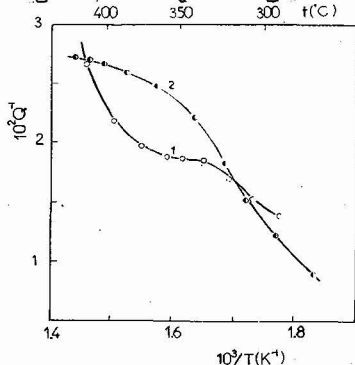


Fig. 3. Internal friction curves of 99.999 aluminium single crystal: Curve 1. Measured in ascending temperatures. Curve 2. Measurements started at 435°C with descending temperatures.  $f = 1.8$  Hz.

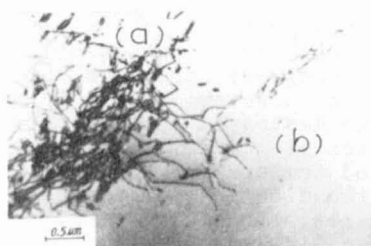


Fig. 4. Dislocation configuration in 99.999 aluminium single crystal after heating up to 435°C.

III. 3. The single-crystal specimen exhibiting the "365°C peak" was deformed 3% at room temperature and then annealed at 600°C for 2 h. The internal friction was measured with descending temperatures and the 365°C peak disappeared as shown in Fig. 5. TEM observations on the

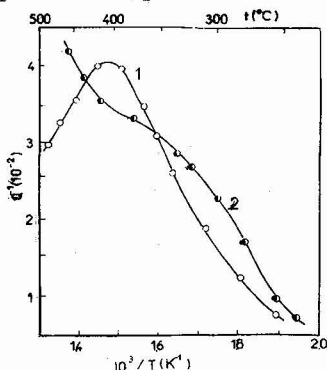


Fig. 5. Internal friction curves of 99.999 aluminium single crystal: Curve 1. Annealed at 600°C for 2 h. Curve 2. Stretched 3% at room temperature and then annealed at 600°C for 2 h.  $f = 1.8$  Hz.

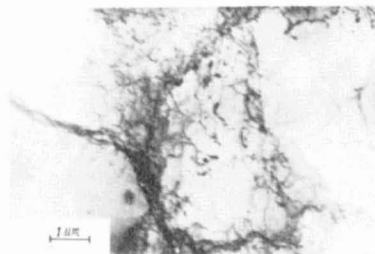


Fig. 6. Dislocation configuration in 99.999 aluminium single crystal stretched 3% at room temperature and then annealed at 600°C for 2 h.

film prepared from this specimen showed that the dislocation density increases considerably to exceed  $10^{10}/\text{cm}^2$  with the formation of cell structures having an average size of several  $\mu\text{m}$  as is shown in Fig. 6. The dislocations are concentrated at the walls of the cell. The misorientation angle between the cell structures was found to be about  $0.1^\circ$ .

IV. Discussions - Conclusions can be made according to the result of internal friction measurements and TEM observations that the "365°C peak" appeared in 99.999 wt % aluminium single crystal is correlated to the existence of the spatial network wove together by dispersive dislocations in three-dimensional crystal lattice. This peak is suppressed by the appearance of cell structures when the single crystal specimen was subjected to 3 % stretching at room temperature.

The proportion of the spatial network and the cell structure determine the appearance and disappearance of the 365°C peak and the height of the peak. The 365°C peak only appears after the single crystal specimen prepared by dynamic annealing method was annealed at an elevated temperature. Consequently, an annealing at an elevated temperature is a necessary condition for the formation of this stable spatial network. However, once this stable structure in a single crystal specimen was destroyed by deformation with the formation of cell structures, then it cannot be reformed again simply by an annealing at an elevated temperature. Instead, the high temperature internal friction background beyond 365°C increased with the existence of such cell structures. TEM diffraction contrast analysis showed that some of the dislocation segments composing the spatial network are situated on high index planes. This indicates that dislocation climb is the principal process in forming the spatial network.

#### References

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