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"Recent Research on Mechanical Properties and the Structure of Grain Boundaries"

"The Appraisal and the Mechanism of the Bamboo Boundary Internal Friction Peak" (Part I)

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Recent Research on the Mechanical Properties and the Structure of Grain Boundaries

I. The Appraisal and the Mechanism of the Bamboo Boundary Internal Friction Peak

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Abstract

In the beginning of the eighties, an internal friction peak situated below the optimum temperature of the grain boundary peak of fine-grained specimens (the Kê peak)was observed in high-purity aluminium consisting of bamboo crystals. This discovery has given rise to much controversy and opinion has been expressed that this peak is only the Kê peak shifted toward lower temperature and is not a new peak. However, our systematic study showed that the behavior of this peak is quite different from that of the Kê peak.

Recently, the bamboo boundary internal friction peak and the Kê peak were observed separately on one internal friction curve in sheet specimens consisting of mixed grains. This shows definitely that the bamboo boundary peak is a new peak. TEM observations showed that dislocation substructures exist nearby the bamboo boundaries. Accordingly we propose that such substructures intersecting and interacting with the bamboo boundaries are dragged along with the bamboo boundary during the viscous sliding of the boundary, so that the sliding process is limited with the appearance of an internal friction peak.

A four-parameter mechanical model was suggested for describing the relaxation behavior of the bamboo boundary peak. The mathematical formulations and the experimental conditions for the appearance of the bamboo boundary peak derived according to this model are in accord with experiments.

1. Introduction

Research on the property and structure of grain boundary in metals is an old but unsolved problem. As grain boundaries exist in almost all the metals and alloys in practical use and have a crucial effect on their properties, the research on grain boundaries has indisputably an academic as well as practical significance.

For many years, the relationship between the property and the structure of grain boundary has been studied with various experimental techniques. In 1947, Kê [1] reported his results of research on the behavior of the grain boundaries in 99.991 polycrystalline aluminium with anelastic measurements which include the measurement of the variation with temperature of the internal friction and elastic modulus and the micro-creep under constant stress as well as the stress relaxation under constant strain. An internal friction peak versus the temperature of measurement was observed with an optimum temperature of 285℃ when the frequency of vibration is 0.8Hz. On the basis of a series of measurements and correlative theoretical analysis, Kê considered that this internal friction peak which was absent in single crystal specimen, is associated with the stress relaxation across grain boundaries and named it the grain boundary internal friction peak. These results show that the grain boundary behaves in a viscous manner, i.e., it has a coefficient of viscosity varying with temperature. Considering the metal containing grain boundaries as a single entity, this viscous behavior is different from that exhibited by a liquid, since the viscous sliding under-taken along the grain boundary under applied shearing stress is limited. This is because that the grain edges and corners existing in polycrystalline specimens exert a blocking effect on the viscous slide along the boundary, so that the slide cannot proceed unlimitedly. In other wordes, the stress applied across the boundary is relaxed through the sliding along the boundary and the relaxed stress concentrates at the regions around the grain edges and corners causing an elatic distortion there. This elastic distortion produces a counter stress prohibiting the boundary sliding so that the sliding stops eventually. As such, a grain boundary internal friction peak can be observed expercimentally, and a saturated value for the micro-creep, the shear modulus relaxation is obtained eventually with a definte value of the relaxation strength. This conclusion was subsequently confirmed by experiments of numerous research workers and serves as the foundation of the viscous sliding model of grain boundary[2].

2. The controversy over the origin of the grain–boundary internal friction peak

In 1976, Gondi and co–workers[3] measured the internal friction of single crystal aluminium (99.6%) and an internal friction peak appeared after the specimen had been slightly cold–worked. They concluded that the position of the peak is identical to the peak observed by Kê in polycrystalline 99.991 aluminium. Consequently, they consider that the so–called grain–boundary internal–friction peak is not originated from grain boundary process but is, on the contrary, contributed by the dislocations inside the grains. So they named the classical grain–boundary peak the Kê peak. At about the same time, Woirgard et al.[4, 5] also reported that a feeble internal–friction peak appeared in single crystal aluminium (99.99%), and the position of the peak is close to that of the grain–boundary peak observed by Kê, so that they considered also that the "Kê peak" is not originated by grain boundary process. A close analysis on the experiments of Woirgard et al. showed that the single–crystal specimen they used may contain a small amount of fine crystals and also that their specimens were slightly cold–worked.

In the paper presented at ICIFUAS–7, Riviere, Amirault and Woirgard[6] claimed once and again that the peaks associated with the poly and the single crystals are identical as regards the location and the behavior during successive annealings. So the authors asserted that it is confirmed that the geometry of the dislocation arrangment has the most important influence to these peaks and the grain boundaries have only an indirect influence through the interactions with the dislocation network. In commenting this paper, Kê [7] expressed the following opinions: The experimental results reported by the authors in this paper and in those published previously[4, 5] claiming that the relaxation peaks appeared in slightly deformed single crystals are in the same temperature range as the "orthodox" grain boundary peaks in polycrystals need to be re–examined and analysed carefully and the assertion that the grain boundary peak is due to the disloction motion inside the grains has to be critically discussed. It seems that, in many cases, the authors compared the results of polycrystalline and single–crystal specimens with different purities, or with different cold–work states. Although the internal friction they observed sometimes occur in nearly the same temperature region in polycrystalline and single–crystal specimens, but actually they are not identical by careful analysis. The procedure adopted for substracting the high–temparature internal friction background should be examined with excreme care. Also the height of the peak in polycrystalline specimens is much higher than those in the single–crystal specimens. This leads to the question that whether the single-crystal specimens they used are crystals without any grain boundaries.

To be sure, relaxation peaks may also appear in single crystals when deformed. It is very difficult to avoid the cold-working effect introduced in mounting the highly softened single-crystal specimens for internal friction measurements.

It is certain that the cold-work state and the dislocation configuration in the grain proper would influence the grain boundary behavior and the dislocation configuration in the grain boundaries, but the grain boundary peak observed in polycrystals needs not to be associated with the same dislocation group as that responsible for the possible relaxation peaks observed in slightly deformed single crystals. Thus it seems premature to consider that the "orthodox" grain boundary peak observed in completely recrystallized polycrystalline specimens is not originated from grain boundary relaxation process.

In order to clarify the questions mentioned above, efforts have been made by Kê, Cui and Su[8] to compare the internal friction of polycrystalline and single-crystal aluminium specimens of the same purity and to ensure that the single-crystal specimen used does not contain any fine crystals or bamboo grain boundaries and the specimen had not been cold-worked. Extensive experimental results showed that in genuine aluminium single crystals prepared by three different procedures (static annealing method, dynamic annealing method, growth by zone-melting) without being deformed, no internal-friction peak appeared around the temperature region of the grain-boundary internal friction peak (Kê peak) appeared in polycrystalline aluminium of the same purity (99.991 and 99.999% Al). Consequently the grain boundary peak observed by Kê in polycrystalline specimens cannot be attributed to the presence of dislocations inside the grains and can only be attributed to the precess taking place at the grain boundaries.

During an extensive study on the classical grain boundary internal friction peak (Kê peak), two types of peak were observed nearby the temperature region of the Kê peak[8, 9, 10]. One type of peak appears in macrocrystalline or bamboo crystalline specimens at a temperature about 260℃ [9, 10] which is lower than that of the Kê peak and maybe named the macro-crystalline boundary peak or bamboo boundary peak. The other type of peak appears in single crystals subjected to cold-work, especially by twisting deformation, at about 360℃ which is higher than that of the Kê peak[11, 12]. It is evident, thus, that the controversy over the origin of the Kê peak is due to the mis-identification of the peak appeared in single crystals when cold-worked or of the peak appeared in macrocrystalline (bamboo-like) specimens as the Kê peak.

To make the issue clearer, let us put up the following criterion for defining the fine–grained polycrystalline, macro–crystalline and bamboo–grained specimens. A fine–grained specimen is meant that all the grains are smaller than the diameter of a wire specimen or the thickness of a sheet specimen. A macro–crystalline specimen is meant that some of the grains are larger than the diameter or the thickness of the specimen. The bamboo–grained specimen is a special kind of macro–crystalline specimen, in which the grain boundaries extend all the way across the diameter of a wire specimen and all the boundary planes are perpendicular to the axis of the specimen. It will be shown below that the grain boundaries in different kinds of specimens behave differently, so that one should be aware of what kind of specimen is concerned when the observed internal friction peak is discussed.

3. The appraisal of the bamboo boundary internal friction peak

The appearance of a bamboo boundary internal friction peak is highly puzzling. In a wire specimen consisted of bamboo boundaries, all the boundaries are perpendicular to the axis of the specimen. No grain edges and corners exist in such a specimen as in the case of a fine–grained polycrystalline specimen, so that the boundary sliding under torsion (shear) stress should not be blocked and the sliding should proceed unlimitedly and the grain boundary internal friction peak should not occur. Experiments as well as theoretical analysis show that the irregular regions or the ledges possibly existing along the boundary cannot effectively block the macroscopic viscous sliding of the boundary at high temperatures. Such ledges will be overwhelmed by boundary sliding during the first half–cycle of vibration in internal friction measurement and cannot give rise to a counter stress so as to recover the original state of the boundary during the opposite half–cycle of vibration. Thus if an internal friction peak does appear in a specimen consisted of bamboo boundaries, then the foundation of the viscous sliding model of grain boundary will be in doubt and constitutes a serious challenge to the experimental results and theoretical analyes obtained by numerous researchers in this field in the last forty years.

Before introducing the systematic study performed in our laboratory for giving a definite answer to this problem. It is important to point out that many of the internal friction peaks observed by various authors in cold–worked or annealed pure aluminium around an intermediate temperature are actually the bamboo boundary peaks or are superposed with the bamboo boundary peak. These include the 177℃ internal friction peak observed by Esnouf et al. and No et al.[13–16] in cold–worked 99.9999 aluminium which was considered to be connected with the movement of lattice dislocations and the internal friction peaks observed by Iwasaki[17] in annealed 99.999 aluminium around somewhat higher temperatures. The average grain size in the specimens they used is already larger than the diameter of the wire specimen or is larger than the thickness of the sheet specimen although they did not mention this explicitly.

In order to clarify this situation, Cheng and Kê[18] prepared a series a specimens of zone–melted 99.9999 aluminium containing various numbers of bamboo grain by static or dynamic annealing method.

Each specimen was annealed in–situ of the torsion pendulum apparatus at 600℃ for 2h and internal friction was measured at descending temperatures with a frequency of vibration of 1.15Hz and a maximum excitation strain amplitude of 10^{-5}. Fig.1. shows the relationship between the height of the internal friction peak (Q^{-1}_{max}) and the number of bamboo boundary N containing in a specimen of 10cm length. It is seen that Q^{-1}_{max} is directly proportional to N and the line passes through the origin, showing that the peak is definitely associated with the presence of bamboo boundaries. This result is identical to that obtained by Kê et al. for 99.999 aluminium reported previously[19].

Fig.1. The relationship between Q^{-1}_{max} and N.

The optimum temperature of the internal friction peak corresponding to various N is shown in table 1.

Table 1. Relaxation parameters of the bamboo boundary peak corresponding to N

N(per 10cm)	8	16	18	28	36
T_p (℃) at 1Hz	169	167	184	158	150
Q^{-1}_{max}	0.0180	0.0346	0.038	0.0612	0.0690

It is seen that the average T_p for these five bamboo–grained specimens is 167℃ which is close to the value of 177℃ observed by Esnouf et al. in cold–worked 99.9999 Al.

The effect of cold–work on the bamboo boundary peak in 99.9999 aluminium was also studied symmetrically by Cheng and Kê[8]. The results (Fig.2) show that the internal friction peak previously observed in cold–worked 99.9999 aluminium by Esnouf et al. and No et al.[13–16] and claimed to be due to lattice dislocations is actually a composite peak consisting of three sub–peaks: P_H, $P_B$and P_L peaks, where P_B is the bamboo boundary peak. P_H and P_L and two peaks originated from cold deformation and are situated, respectively, at higher and lower temperatures than that of P_B.

Fig.2. Effect of twisting deformation on the bamboo boundary peak of a 99.9999 aluminium specimen having N = 8 ± 1 (in 10cm). Specimen twisted to 0.33% and then annealed at various temperatures. Annealing temperatures for curves 1, 2, 3 are, respectively, 400, 460 and 600℃ for 2h. Curve 1′ is the modulus curve corresponding to curve 1. Curve 1″ is the $\partial f^2 / \partial (10^3/T)$ curve.

A specimen having N = 8 ± 1 (in 10cm) was twisted to 0.33% at room temperature and then annealed at various temperatures in situ of the torsion pendulum apparatus. When annealed at 400℃ for 2h, the internal friction curve is shown by curve 1 of Fig.2. The shape of the curve is quite complicate, showing that it consists of several component peaks. The corresponding modulus curve (f^2) is given by cruve 1′. Curve 1″ is the curve representing the differential of f^2 with respect to $10^3/T$. It is seen that there are three peaks on this curve situated at 223, 185 and 111℃, respectively, corresponding to P_H, P_B and P_L.

When the specimen was annealed at 460℃ for 2h, the internal friction curve becomes less complicate as is shown by curve 2. After an annealing at 600℃ for 2h, only the P_B peak remains as shown by curve 3. This shows that the effect introduced by twisting at room temperaure was completely eliminated by an annealing at 600℃. On the other hand, experiments show that the appearance of P_H and P_L is more dominant in cold–worked specimens with larger bamboo grains under otherwise similar conditions.

The experimental results give above show difinitely that a bamboo boundary can give rise to an internal friction peak. Now the question is: what is the difference between this bamboo boundary peak and the fine–grained grain boundary peak (Kê peak).

The following experiments will show that the behavior exhibited by the bamboo boundary peak is quite different from that of the Kê peak for fine–grained specimens.

The early experiments of Kê[1] on 99.991 aluminium showed that the relaxation strength (2 Q^{-1}_{max})associated with the grain boundary peak of fine–grained specimens is independent of the grain size of the specimen provided that the grain size is smaller than the diameter of the wire specimen. In order to compare this characteristic behavior of the fine–grained grain boundary peak (Kê peak) with that of the bamboo boundary peak, internal friction measurments were made by Zhu and Kê[20] with aluminium wire specimens of the same purity (99.999%) in both cases. The diameter of the specimen is 1mm and the frequency of vibration is 0.62Hz.

The specimen was annealed successively at 350, 385, 420, 473℃ for 8h, and at 540℃ for 1h and the grain sizes obtained are 0.2, 0.3, 0.4, 0.6 and 0.9mm, respectively. The Q^{-1}_{max} of the corresponding internal friction peaks (with the high– temperature internal friction background subtracted) are 0.069, 0.070, 0.065, 0.070 and 0.068 respectively as shown by curve 1 in Fig.3. The grain size dependence of Q^{-1}_{max} in the case of the bamboo grain boundary peak in 99.999 aluminium previously determined [19] is shown by curve 2 for comparison. It is seen from Fig.3 that the grain size dependence of Q^{-1}_{max} for the Kê peak and the bamboo boundary peak is remarkably different.

Fig.3. The relationship between Q^{-1}_{max} and the grain size in the case of Kê peak (1) and the bamboo boundary peak (2). The diameter of the wire specimen is 1 mm.

The difference shown in Fig.3 may be explained as follows. The energy dissipated during the sliding along the boundaries may be measured by the product of the relative displacement across the boundary and the shear stress resisting the displacement. In the range around the optimum temperature of the internal friction peak, since the shear stress is constant, the internal friction is proportional to the grain boundary area per unit volume multiplied by the average relative displacement across a grain boundary. For fine–grained specimens with grain edges and corners, the relative displacement is directly proportional to the grain size d but the grain–boundary area per unit volume is inversely proportional to d, so that the energy dissipation is independent of d. For bamboo–grained specimens, the relative displacement is independent of the average distance between bamboo boundaries L, while the grain–boundary area per unit volume is inversely proportional to L, so that the internal friction is inversely proportional to L or directly proportional to N.

Quantitative study was made by Zhu and Kê[20] on another characteristic behavior of the bomboo boundary internal friction peak which is different with that of the Kê peak. This concerns in the shift of the peak with an increase of annealing temperature. Two sheet speciemns having the same width (4.0mm) and length (9.0cm) but with thickness 1.0 and 0.5mm, respectively, were prepared by cold–rolling through the same percent of reduction. They were successively annealed in situ in the torsion pendulum apparatus at various temperatures and internal friction measurements were taken at descending temperatures. Experimental results show that the effect of annealing temperature

T_a on the optimum temperature T_p of the bamboo boundary peak can be classified into three stages as T_a becomes successively higher (Fig.4). (i) T_p tends to shift toward higher temperature. (ii) T_p shifts rapidly toward lower temperatures. (iii) T_p shifts slowly toward lower temperatures and eventually becomes stabilized. Metallographic examination on the grain size of the specimens shows that in the first stage, the specimen was recrystallized and the grain size is much smaller than the thickness of the specimen. In the second stage, some of the grains grow beyond the specimen thickness at first, then most of the grains grow beyond so that the grain edges and corners originally spread over the width of the specimen disappeared completely. In the third stage, all the grains of the specimen become stabilized bamboo grains.

Fig.4. Variation of T_p with T_a for a) 0.5 mm and b) 1.0 mm sheet specimen.

Similar experiments were made with four wire speciemns with diameters 0.7, 1.0, 1.2 and 1.5mm prepared by cold-drawing to the same percent reduction of area (75% RA) but were then reduced to the relevant diameters by chemical etching. The effect of increasing T_a on T_p for each specimen can also be classified into three stages. The results for the 0.7mm wire specimen are shown in Fig.5. Metallographic examination shows that the critical annealing temperature of T_a for the beginning of the second stage is just the temperature at which the grains in the specimen grow larger than the diameter of the wire with the disappearance of grain edges and corners.

Fig.5 Variation of T_p with T_a for specimen with diameter of 0.7mm.

Similar results have been observed by Kê et al.[9, 10] with a specimen of given diameter (1 mm) where the grain size was increased until exceeding the specimen diameter by successive annealing at higher temperatures.

In order to examine the influence of the mode of vibration in internal friction measurement on the observed effect described above, compact spring specimens were used for similar studies with a vibration mode of push-pull bending. Similar results were observed as in the cases of sheet and wire specimens measured with a shearing vibration mode. These results demonstrate quantitatively and conclusively that the different shift behavior of the bamboo boundary peak with T_a from that of the Kê peak is common to sheet, wire and compact spring specimens when internal friction is measured in torsional or bending vibration modes. As such, we can conclude that the bamboo boundary peak is a different peak from the Kê peak.

4. The controversy over the origin of the bamboo boundary peak

In 1983–1985 Iwasaki[17] reported that the height of the internal friction peak measured with a bending pendulum for an aluminium sheet specimen containing only one grain boundary (cut off from a large sheet containing very large grains) is approximately proportional to the resolved shear stress acting along the grain boundary irrespective of the type and the nature of the boundary. Accordingly he suggested that the internal friction peak appeared in a specimen containing only one boundary is due to the sliding along the boundary. Recently, Iwasaki[21] argued that the bamboo boundary is one type of grain boundares, the bamboo boundary peak is one kind of grain boundary peak, so that in this sense there should be no distinction between the Kê peak for fine–grained specimens and the peak for macro–crystalline or bamboo–grained specimens. The main point is thus whether the transition from the Kê peak to the bamboo boundary peak really occurs or not, and whether the origin of both peaks are the same or not. As to the controlling factor for the sliding along bamboo boundary, Iwasaki emphasizes that "the surface of the aluminium specimen is usually covered with a strong Al_2O_3 layer which constrains the sliding of the bamboo boundary just as the triple point of the conventional grain boundaries in the fine–grained specimen does. The same peaking mechanism is then possible to operate regardless of the grain size or charactor". In order to examine the influence of a surface layer on the appearance of the macro–crystalline peak, systematic experiments have been carried out by Kê et al.[22] by correlating internal friction measurements with Auger spectrocopic analysis. Curve 1 of Fig.6 gives the internal friction curve of a macro–crystalline 99.999 aluminium apecimen after the Al_2O_3 surface layer was scraped by argon ion thinning technique. Auger analysis showed definitely that the aluminium matrix has been exposed. The internal friction measurements were taken in a computerized inverted torsion pendulum under a vacuum of 4×10^{-5} Torr. The macro–crystalline peak appears around 225℃ with f = 1.2Hz. Curve 2 of Fig.6 gives the internal–friction curve of a macro–crystalline 99.999 aluminium specimen annealed at 600℃ for 1h in air so that a thick Al_2O_3 layer was formed on the surface of the specimen. It can be seen on comparing curves 1 and 2 that there is no noticeable difference between these two extreme cases.

Fig.6 Effect of Al_2O_3 surface layer on the macro-crystalline peak is 99.999 aluminium: (1) without Al_2O_3 surface layer. (2) with a thick Al_2O_3 surface layer.

As has been described above, a distinction between Kê peak and the macro-cystalline peak is their different response toward annealing treatment. The former shifts to higher temperatures while the latter shifts toward lower temperature with increasing annealing temperature. However, Iwasaki argues that the shift of the macro-cystalline peak toward lower temperature was observed only after creep deformation. It seems that this conclusion cannot be true in general. In order to repeat Iwasaki's results, internal friction measurements were made with the Japanese-made 99.999 aluminium used by Iwasaki. The effect of annealing temperature on the position of the macro-crystalline peak is shown in Fig.7[22]. It is seen that the peak shifts definitely toward lower temperatures with increasing annealing temperature (without creep deformation).

Fig. 7. Effect of annealing temperature on the position of the macro-crystalline peak of 99.999 aluminium (Japanese-made): (1) 450℃ anneal for 2h, grain size 1.1mm. (2) 500℃ anneal for 2h, grain size 2.2mm. (3) 600℃ anneal for 2h, grain size 2.5mm. Diameter of specimen 1mm.

On the other hands, experimental results recently obtained in our laboratory[22] on the effect of creep deformation on the macro-crystalline peak of 99.999 aluminium are shown in Fig.8. Curves 1 to 3 show, respectively, the cases of no creep deformation, 1.28% and 2.83% creep deformation at 495℃. It is seen that although some changes occurred among the three curves, the shift of the internal friction curve toward lower temperatures because of creep deformation is not evident.

Fig.8. Effect of creep deformation on the macro-crystalline peak of 99.999 aluminium: (1) without creep deformation. (2) 1.28% creep deformation at 495℃. (3) 2.83% creep deformation at 495℃.

Considering that Iwasaki's results are, as he stated himself, due to the effects of the stresed annealing caused by the excess counter weight added to the balancing one of the inverted torsion pendulum, and that the creep deformation at 450℃ was as high as 3.9% with an excess counter weight of 200g[17], the shift of the bamboo peak toward lower temperatures because of creep deformation may occur only when the amount of deformation is quite large. In order to examine whether this is really the case, Zhu and Kê[20] performed the following experiment. The wire specimen used contains 10 bamboo grains with an average length of 1 cm. Marker lines almost perpendicular to the bamboo boundaries were scribed on the specimen surface ofr sliding measurements. The specimen was then annealed in situ of the torsion pendulum apparatus at 630℃ for 0.5h and an internal friction peak appeared when measurements were taken at descending temperatures. The specimen was then twisted counter-clockwise at 620℃ in-situ of the torsion pendulus apparatus to 30°. This corresponds to an average shear displacement of 4μm across each bamboo boundary with a shear stress of about 50MP_a. The bamboo boundary internal friction peak shifts toward lower temperatures after this twisting. When the wire was twisted 30° clockwise back to its original position, the peak continues to shift toward lower temperatures. It shifts more after a further twisting of 30° clockwise. However, the height of the peak remains almost unchanged after these twistings (with the high-temperature internal friction background subtracted).

Optical microscopy reveals that the boundaries perpendicular to the specimen axis have all slid to a certain extent after twisting. This demonstrates that the in-situ twisting inside the torsion pendulum apparatus gave rise to grain boundary sliding. Consequently the shift of the bamboo boundary peak toward lower temperatures is associated with the effect of grain boundary sliding.

To sum up, internal friction measurements show that the bamboo boundary peak may shift toward lower temperatures through two procedures performed on the specimen before internal friction measurement: (1) by high temperature annealing of a bamboo-grained specimen; (2) by a relatively large creep deformation or grain boundary sliding subjected to the specimen.

In the early work of Iwasaki concerning the high-temperature internal-friction peak of 99.999 aluminium, the specimens used are bamboo-grained since the grain size is larger than the specimen diameter. He considered that the "main" internal-friction peak he observed is composed of two components[17]: the high temperature component and the low temperature component. The former component is prominent in the fully annealed state and the height of the peak is related to the grain size, so that this peak is the conventional grain-boundary peak (Kê peak). We consider that this high temperature component is actually the macrocrystalline or bamboo boundary peak and the lower temperature component which is prominent in the deformed state is related to lattice dislocations as Iwasaki suggested. This becomes evident considering the experiments of Cheng and Kê[18] as has been described in § 3 above (cf. Fig.2).

In Iwasaki's internal-friction measurement with bending pendulum on 99.999 aluminium sheet specimens (0.8 mm thick) containing only one grain boundary with orientation angle of 0, 45, 90 ° with the axis of the specimen[17], it was found that the high-temperature component of the main peak is prominent only when $\theta=45°$ where the resolved shear stress along the boundary is the largest. This result indicates that the underlying process associated with the high-temperature component of the main peak is the sliding along the boundary similar to the case of the Kê peak. However, the boundary concerned here is evidently the bamboo boundary, since only one boundary exists in the specimen. Accordingly it is not feasible to claim that this high temperature component of the main peak is just one type of Kê peak and has the same origin as the Kê peak. The mechanism of the macrocrystalline or bamboo-boundary peak will be discussed later in § 6 after we demonstrated in § 5 that the bamboo boundary peak is really a new peak and has a different mechanism as the Kê peak.

5. Conclusive evidence affirming that the bamboo boundary peak is a new peak

Numerous experiments have shown that the characteristic behaviors exhibited by the bamboo boundary internal friction peak are markedly different from those of the Kê peak, so that it should be considered as a new peak. However, there are, up to the present, no experimental results demonstrating that these two peaks are two separate peaks. Opiminions have thus be expressed[21] that the bamboo boundary peak is originated from a shift of the Kê peak toward lower temperatures. In 1989, Kê and Zhu[23] reported their effort of separating these two peaks with sheet specimens guided by the following considerations. The stress distribution across the diameter of a wire specimen prepared by cold-drawing is isotropic. Consequently the recrystallized grains formed after annealing are rather regular and grow uniformly during further annealings. The transition of the grain size from being smaller to being larger than the diameter of the specimen is gradual and continuous, so that during the transition from the Kê peak to the bamboo boundary peak the two peaks are superimposed and appear as a composite peak. They can be observed individually and separately only when one type of grains, either fine-grains or bamboo grains exist in the specimen. Contrary to this situation, the stress distribution in sheet specimens prepared by cold-rolling is anisotropic. The recrystallized grains formed after annealing are not uniform, and mixed grains (smaller or larger than the thickness of the specimen) may exist in the specimen. As such, we anticipate that in this case, the bamboo boundary peak may be separated from the Kê peak.

A 3mm rod of 99.999 aluminium rod produced in China was cold–rolled to sheet specimens with dimensions of $100 \times 4.2 \times 1mm^3$. The specimen was annealed in–situ of the torsion pendulum apparatus and internal friction was measured with descending temperatures. The frequency of vibration is 1.5Hz and the maximum strain amplitude is 1.2×10^{-5}. Only a small normal amplitute effect (less than 10%) was observed.

The internal friction curves after the specimen was annealed at various temperatures are shown in Fig.9. Curve 1 of Fig.9 corresponds to an annealing temperature of 445℃ for 1.5h. the P_1 peak appearing at 310℃ is evidently the Kê peak. An annealing at 530℃ for 3.5h gives curve 2. The internal friction peak shifts to 314℃ and the high–temperature internal friction background is considerably reduced. At the same time a small hump appears on the lower temperature branch of the internal friction peak. Curves 3 and 4 correspond to annealings at 610℃ for 1.5h and 640℃ for 2.5h, respectively. It is seen that the high–temperature background is further reduced are the width of the peaks increases considerably. Both peaks are unsymmetrical and the low–temperature branch of each peak drops down much slower than the high–temperatue branch. This indicates clearly that a new peak situated at a lower temperature than P_1 is superposed on P_1.

Fig.9. Internal friction curves of cold–rolled sheet specimen annealed at various temperatures. Curves 1 to 4 correspond to annealing temperatures of 445, 580, 610 and 640℃.

In Fig.10 are shown the curves of Fig.9 with the high–temperature background subtracted. Curve 4′ of Fig.10a corresponds to curve 1 of Fig.9. It is seen that this curve which was plotted against 1 / T, is very symmetrical. Consequently it is a single peak of Debye type and is identified as the Kê peak (peak P_1). Curve 2′ can be resolved into two symmetrical peaks: P_1 and P_2 where P_2 is a new peak appearing at a temperature lower than that of P_1. The height of P_1 is lowered with the simultaneous appearance of P_2. The resolution of curves 3′ and 4′ into P_1 and P_2 is shown in Fig.10c and d in which the height of P_1 becomes progressively lower indicating that the proportion of fine grains in the specimen becomes progressively smaller. The progressive increase of the height of P_2 indicates that the proportion of bamboo grains becomes progressively larger.

Fig.10. Resolution of the internal friction curves a) 1, b) 2, c) 3 and d) 4 of Fig.9.

Metallographic observations were made on the surface of the sheet specimens (1 mm thick) annealed at various temperatures after internal friction measurements. The metallographs are shown in Fig.11a, b, c. In Fig.11a for which the specimen was annealed at 530℃, only a few of the grains run across the thickness of the specimen. This is the state of co–existence of fine grains and macrocrystalline grains (bamboo grains) with fine grains dominating. The corresponding internal friction peaks P_1 and P_2 are shown in Fig.10b, and their heights are 0.052 and 0.008 respectively.

Fig.11. Metallographs taken on the surface of cold–rolled sheet specimen annealed at a) 530℃ (×25), b) 610℃ (×45), c) 640℃ (×25).

In Fig.11b (610℃ anneal), most of the grains run across the thickness of the specimen. Fine grains appear only in some localized regions. The corresponding peaks P_1 and P_2 are shown in Fig.10c and their heights are, respectively, 0.025 and 0.026.

In Fig.11c (640℃ anneal), almost all the grains run across the thickness of the specimen, and the P_1 and P_2 peaks are shown in Fig.10d with heights 0.010 and 0.038 respectively.

The co–existence of P_1 and P_2 peaks described above has been observed repeatedly in several sheet specimens of 99.999 aluminium and the relative heights of P_1 and P_2 conform to the relation proportion of fine grains and bamboo grains existing int he specimen. Consequently we are convinced that P_1 and P_2 are two separate peaks with different origins. The P_1 peak is associated with fien–grianed grian boundaries (Kê peak) and the P_2 peak is associated with bamboo boundaries.

6. Mechanism of the bamboo boundary internal friction peak

The appearance of an internal friction peak signifies that the relaxation process associated with the peak is limited. It is well–known that the factor limiting the grain boundary in fine–grained specimens is the existence of grain edges and corners. Since such grain edges and corners are absent in bamboo crystals, it is puzzling what is the factor limiting the relaxation process across bamboo boundaries. Similarity of the activation energy associated with both peaks (1.4 eV)[19, 18, 1] suggests that the mechanism of the relaxation process across bamboo boundaries is also the viscous sliding along grain boundaries as has been established in the case of fine–grained grain boundaries.

Ogino and Amano[24] suggested that the boundary irregularities such as boundary ledges may act as obstacles and play a similar role as the triple points at the grain–boundary joints in fine–grained specimens. Such ledges can be formed as a result of the coalescence of grain boundary dislocations during grain growth and also by the penetration of lattice dislocations. However, because of the climbing rate of grain boundary dislocations is very high, such ledges cannot block the macroscopic viscous sliding along the boundaries. Consequently such ledges will be overwhelmed by boundary slidng during the first half–cycle of vibration in internal friction measurement and cannot give rise to a counter stress so as to recover the original state of the boundary during the opposite half–cycle of vibration. Fujita[25] pointed out that in the case of bamboo crystals the boundary plane is deformed to a complex shape by internal stress induced by pile–up dislocations. Such irregular dislocation configurations nearby the bamboo boundaries may play a somewhat similar role as the grain edges in fine–grained specimens. Transmission electron microscopy studies by Kokawa, Watanabe and Krashima[26] provided evidences showing that a boundary in 99.999 aluminium coarse–grained polycrystals is intersected by a sub–boundary after a small amount of sliding during creep deformation at 427℃ under a stress of 2 MP_a. It seems that dislocation substructures always exist near the boundary after sliding. Rhines et al.[27] showed by X–ray Laue analysis that dislocation substructures of very high density exist in the grain boundary area in pure aliuminium after grain boundary slidng. If this is due to the absorption of lattice dislocations toward the boundary during boundary sliding, then it is conceivable that numerous lattice dislocations have been drawn to the vicinity of bamboo boundaries during the preparation of the bamboo–crystalline specimen by the method of dynamic or static strain annealing. This is to say that dislocation substructures have already existed in the vicinity of the boundaries in the as–prepared bamboo–crystalline specimen.

In order to find out whether this can happen, transmission electron microscopy studies were made by Zhu and Kê[20] on 99.999 aluminium bamboo specimens prepared by successive high–temperature annealing of heavily cold–rolled sheets. Examples of dislocation substructures existing in the vicinity of grain boundaries are shown in Fig.12.

Fig.12. a) Cell structures near bamboo boundary (450℃ annealing) (15000X). b) Sharpened cell structures near bamboo boundary (600℃ annealing) (20000X). The bamboo boundary is shown by the arrow.

Fig.12a shows the case when the annealing temperature after cold–rolling is low (450℃), in which the dislocation substructures observed nearly the boundaries are principally in the form of cell structures. Fig.12b shows the case when the annealing temperature after cold–rolling is high (600℃), in which the walls of the cell structures are sharpened to become somewhat looser substructures. In Fig.12b the dislocation substructures are seen to intersect the grain boundaries.

It is conceivable that dislocation substructures alreadly exist in the vicinity of bamboo boundaries formed by high–temperature annealing of cold–worked specimens. Subsequent grain boundary sliding or further annealing at high temperatures modifies the configuration of the substructures so that the bamboo boundary peak is shifted toward lower temperatures. As such, we can propose that the bamboo boundary peak results from the appearance of dislocation substructures in the vicinity of bamboo boundaries.

7. The four–parameter mechanical model of the bamboo boundary peak

It is considered that the basic process associated with the bamboo boundary peak is the viscous sliding of the boundary dragging the dislocation substructures (dislocation networks) existed in the vicinity of the boundary. This dragging process controls the viscous sliding along the boundary so that an internal friction peak can appear. However, the limiting action exerted by the dislocation substructures is somewhat different from that offered by the grain edges and corners in the case of fine–grained specimen, which only produce elastic distortion nearby the grain edges and corners. Since the dislocaion is the substructures can move during the boundary sliding, the limiting action is of a relaxation type instead of purely elastic as offered by the grain edges and corners. A four–parameter mechanical model is proposed by Kê and Cheng[28, 29] to describe the relaxation behavior in the case when bamboo boundaries are concerned. This model is different from the three–parameter mechanical model for the relaxation behavior of grian boundaries in fine–grianed specimens.

7.1 Mechanical model for the Kê peak

Fig.13a is a three–parameter mechanical model of Voigt type for fine–grained grain boundaries. The spring (with a force constant k_1) in the lower part (1) represents the grain interior, the dash–pot (with a coefficient of viscosity η_2) in the left side of the upper part (2) represents the boundary propet and spring (with a force constant k_3) in the right side of the upper part (3) represents the grain edges and corners. When a shear stress is applied to the specimen, elastic deformation occurs immediately in the interior of the grains. Since the dash–pot cannot respond to the applied stress at once, the spring in the upper part cannot deform immediately. As time passes by, the dash–pot begins to displace and the stress applied to it starts to relax and is transferred gradually to the grain corners causing elastic distortion there. At the same time the spring starts to deform. When the stress applied to the dash–pot (the grain boundary proper) is completely relaxed, the total stress is transferred to the grain edges and corners. During the removal of the applied stress, the elastic distortion concentrated at the grain edges and corners exerts a counter–stress to the grain boundary so that the boundary slides along the opposite direction and the whole system recovers to its original state. The stress–strain equation derived on the basis of the mechanical model is

$$\tau\dot{\varepsilon} + \varepsilon = \tau J_U\dot{\sigma} + J_R\sigma,$$

in which τ is the relaxation time, σ is the applied stress, ε is the strain, J_U and J_R are the unrelaxed and relaxed compliances respectively. This gives rise to an internal friction peak of Debye type under proper conditions.

Fig.13. Mechanical models describing the relaxation behavior of grain boundaries. a) Fine–grained grain boundaries, b) bamboo grain boundaries.

7.2 Mechanical model for the bamboo boundary peak

In the case of bamboo boundaries, a four–parameter model is suggested as shown in Fig.13b[28, 29]. The only difference between this model and the model shown in Fig.13a is that the right side of the upper part is substituted by a spring in series with a dash–pot. Such a two–parameter model of Maxwell type represents the dislocation substructures intersecting and internacting with the bamboo boundary. The stress–strain equation for this model is

$$\eta_2\eta_3\ddot{\varepsilon} + k_3(\eta_2 + \eta_3)\dot{\varepsilon} = (\eta_2\eta_3 / k_1)\ddot{\sigma} + [\eta_3 + (\eta_2 + \eta_3)k_3 / k_1]\dot{\sigma} + k_3\sigma,$$

where the k's and η's are the force constants and coefficients of viscosity of the springs and dash–pots respectively as indicated in Fig.13b. Since the viscous sliding along the bamboo boundary itself is much eaiser than the movement of the dislocations within the substructures nearby the boundary, we may put $\eta_3 = n\eta_2$ where n is an integer much larger than unity.

It can be shown[28] that the internal friction is

$$\tan\varphi = [m/(1+m)^{1/2}][\omega\tau/(1+\omega^2\tau^2)] + (m/n)[1/(1+m)^{3/2}](1/\omega\tau)[1/(1+\omega^2\tau^2)],$$

where ω is the angular frequency of vibration, $m \equiv k_1/k_3$, $\tau \equiv (\eta_2/k_3)[1/(1+m)^{1/2}]$.

It is seen that the first term on the right-hand side of the above equation represents an internal friction peak of Debye type with a relaxation strength of $m(1+m)^{1/2}$, whereas the second term represents the internal friction background appearing at lower frequencies and increases monotonously with decreasing frequencies.

7.3 Conditions for the appearance of the bamboo boundary peak

In case when $n=\eta_3/\eta_2 \to \infty$, that is, when the dash-pot does not exist, then $\tan\varphi \to [m/(1+m)^{1/2}][\omega\tau/(1+\omega^2\tau^2)]$ and we get the case for the fine-grained grain boundaries. As $m=k_1/k_3$ is much small than unity (the grain interior is much less stiff than the region containing dislocation substractures), we get finally ($k_1=J_U$, $k_3=\sigma J$)

$$\tan\varphi = m[\omega\tau/(1+\omega^2\tau^2)] = \frac{\sigma J}{J_U}[\omega\tau/(1+\omega^2\tau^2)],$$

which is the Debye equation for fine-grained grain boundaries.

Taking $m=0.01$ and $n=1$, 5, 10, 20, 50, 125, 500, 1000, the results of numerical computation giving $\tan\varphi$ for bamboo boundaries as a function of $\ln\omega\tau$ are shown in Fig.14a, b, c. In Fig.14a, the curves for $n=1$, 5 and 10 increase monotonously with decreasing $\omega\tau$ (corresponding to increasing temperatures). Internal friction peaks appear pronouncedly when $n=20$, 50 and 125 as shown in Fig.14b. As n is 500, 1000 or larger, symmetrical internal friction peaks appear as shown in Fig.14c. This gives the bamboo boundary observed experimentally.

Fig.14. Internal friction curves showing $\tan\varphi$ as function of $\ln\omega\tau$ for various values of $n \equiv k_1/k_3$ (cf Fig.13 for the meaning of k_1, k_3 and η_2, η_3).

It is to be noted that the bamboo boundary peak appears only when $n=\eta_3/\eta_2$ is very large. This means that the coefficient of viscosity η_3, the relaxation parameter of the dislocation substructures in the vicinity of the bamboo boundary, must be much large than η_2, the relaxation parameter of the bamboo boundary itself. In other words the dislocation substructures must be less mobile than the viscous sliding along the bamboo boundary under on applied shear stress. Only in such occasion the dislocation substructures interacting with the bamboo boundary can act as an effective factor limiting the viscous sliding of the bamboo boundary.

In the numerical computations given above, we took $m=k_1/k_3=0.01$. This means that the force constant k_1 for the spring representing the interior of the grain should be much smaller than k_3, the fore constant for the spring representing the dislocation substructure. In other words, the mobility $1/k_1$ must be large than the mobility $1/k_2$. This means that the mobility of the dislocation substructures must be smaller than that of the grain interior. This is another condition for the appearance of the bamboo boundary peak.

As the relaxation strength of the bamboo boundary peak is $m[1/(1+m)^{1/2}]$, thus $m=k_1/k_3$ determines the magnitude of the relaxation strength or the height ($Q^{-1}_{max}=m/2$) of the bamboo boundary peak.

7.4 The optimum temperature of the bamboo boundary peak

The optimum temperature of an internal friction peak is determined by its relaxation time τ. For the bamboo bondary peak, we have

$$\tau=(\eta_2/k_3)[1/(1+m)^{1/2}]=\eta_2/k_3=m(\eta_2/k_1)=2Q^{-1}_{max}(\eta_2/k_1).$$

experimental results of Q^{-1}_{max} for 99.999 aluminium are 0.08[26] and 0.002[10], respectively, for fine-grained specimens and bamboo crystal specimens per bamboo boundary). Thus the relaxation time is 0.16 (η_2/k_1) for Kê peak and is 0.004 (η_2/k_1) for bamboo boundary peak. Since η_2 and k_1 are approximately the same for both cases, the relaxation time for the bamboo boundary peak is smaller than that of the Kê peak. Consequenly, the bamboo boundary peak should appear at a lower temperature than the Kê peak. Experiments show that this is always so.

Furthermore, from $\tau=\eta_2/k_3$, we can understand the reasons why the bamboo boundary peak shifts toward lower temperatures by annealing at elevated temperatuers or by grain boundary sliding. It we can assume that the structure of the bamboo boundary itself was not changed by annealing and boundary sliding, the η_2 will remain the same. Thus the shift toward lower temperatures should occur through the enhancement of k_3 and thus the lowering of τ by annealing and boundary sliding. The enhancement of k_3 means that the dislocation substructures nearby the bamboo boundary are more condense and having a stronger interaction with the boundary. It can be seen from Fig.12b that it is so when the specimen was annealed at 600℃. The results of Kokata et la.[26] also show that dislocations intersect pronounced with the boundary after creep deformation.

Expressions of modulus relaxation have also been derived according to the four-parameter model[30], and predications on the basis of numerical computation conform with experimental results.

To sum up, the four parameter mechanical model suggested for the bamboo boundary internal friction peak can describe satisfactorily the role played by the dislocation substructures intersecting and interacting with the bamboo boundaries in constraining the viscous sliding of the bamboo boundaries. This demonstrates the different mechanisms of the bamboo boundary peak and the Kê peak.

8. Concluding remarks

The discovery and the confirmation of the bamboo boundary internal friction peak inspires a new impetus for the research on grain boundary relaxation. Firstly, it becomes obvious that a comprehensive knowledege on the dislocation configuration existed in the close vicinity of the grain boundary as well as the grain boundary structure itself is necessary for a thorough understanding of the mechanism of grain boundary relaxation. Secondly, the peculiar behavior of bamboo boundary relaxation suggests that much pertinent information concerning the structure of grain boundaries can be explored with internal friction and other anelastic measurements. The recent work on the non--linear relaxation behavior observed in as-quenched pure aluminium bamboo-grained specimens may be cited as an example.

Very recently, Kê, Cui and Guan[31] observed an internal friction peak (HT peak) on the high temperature side of the bamboo boundary internal friction peak (BB peak) of 99.999 aluminium. This HT peak appears only in as--quenched but not in furnace-cooled bamboo-grained specimens[32]. Fig.15 shows the case for 650℃ quenching of a specimen containing 45 bamboo boundaries in a length of 10cm[31].

Fig.15. (a) HT peak (curve 5) for 650℃ quenching. (b) Amplitude peaks at the temperature points indicated.

In Fig.15(a), curves 1 and 2 are experimental internal friction curves when the specimen was quenched and furnace-cooled from 650℃ respectively. It is evident that curve 2 is the BB peak. The HT peak (curve 5) was obtained by subtracting the furnace-cooled internal friction curve from the quenched curve after the high temperature background curves 1′ and 2′ had been deducted from curves 1 and 2 respectively. The optimum temperature for the HT peak is 490℃ (frequency 1.4Hz). The activation energy associated with this peak is very high (2.2~ 2.6 eV). The HT peak exhibits pronounced normal and anomalous amplitude dependent effect while the BB peak does not. Fig.15(b) shows the amplitude peaks taken at the temperature points 313, 403, 504, 535 and 590℃ respectively.

It is suggested that the HT peak is connected with the interaction between the dislocation substructure intersecting and interacting with the bamboo boundaries, and the controlling factor to the rate of dragging process is the promoted climbing of the dislocation segments in the substructures by the excess vacancies created by quenching. In the case of furnace–cooled specimen, no excess vacancies exist in the vicinity of the bamboo boundary so that the dislocation segments cannot climb. This is the reason of the different behavior concerning the amplitude effect of the HT and BB peak.

The amplitude effect occurs in the HT peak is originated from the interaction between the jogs during the bowing out (through climbing) of the dislocation segments and the possible shift of the pinning points (the nodes of the dislocation network) at the ends of the dislocation segment under the action of successively higher applied stress or strain amplitude.

A quantitative analysis on the mechanism of bamboo boundaries relaxation awaits further experimental work especially the direct observation on the configuration of the dislocation substructures after annealing at various temperatures and after grain boundary sliding or creep deformation.

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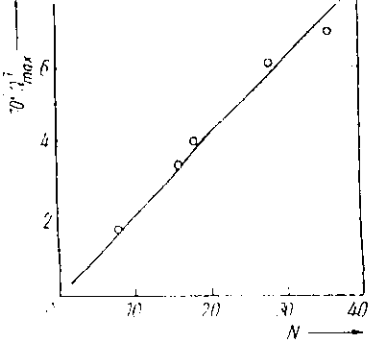
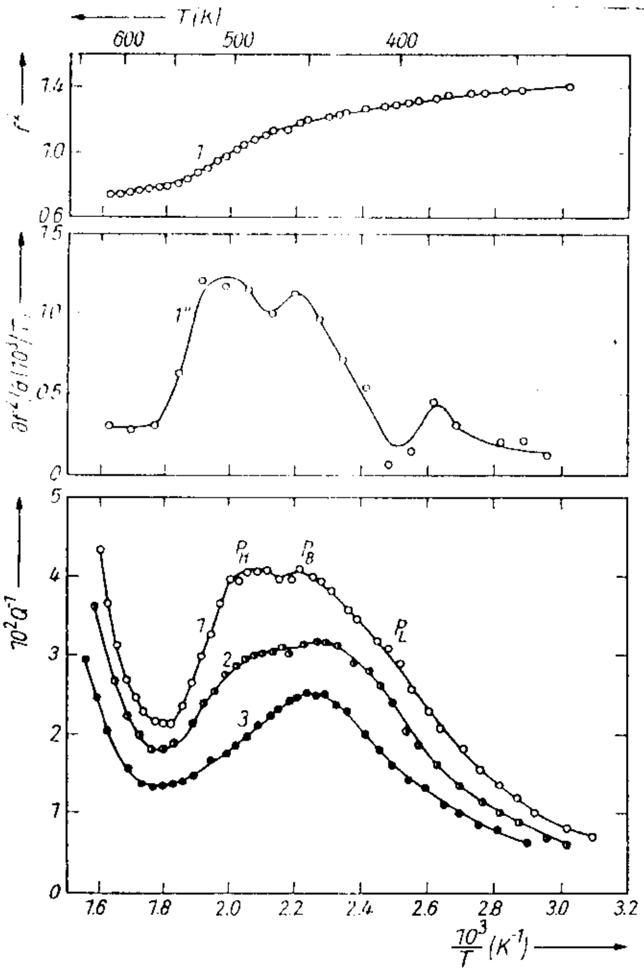
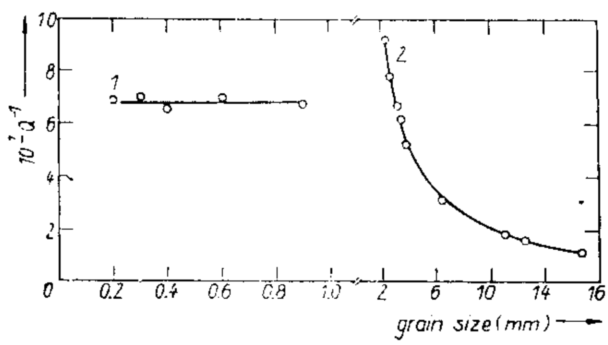
Fig. 1 The relationship between Q_{max}^{-1} and N





Kê. Fig.2

Fig. 2. Effect of twisting on the bamboo boundary peak of a specimen having $N = 8 \pm 1$. Specimen twisted to 0.33% and then annealed at various temperatures. Annealing temperatures for (1), (2), (3) are 673, 733, and 783 K for 2 h. (1′) is the rigidity curve corresponding to (1), (1″) is the curve for $\partial f^2/\partial\,(10^3/T)$



Kê. Fig.3.

Fig. 3. The relation between Q^{-1}_{max} and the grain size in the case of Kê peak (1) and bamboo boundary peak (2). The diameter of the wire specimen is 1 mm

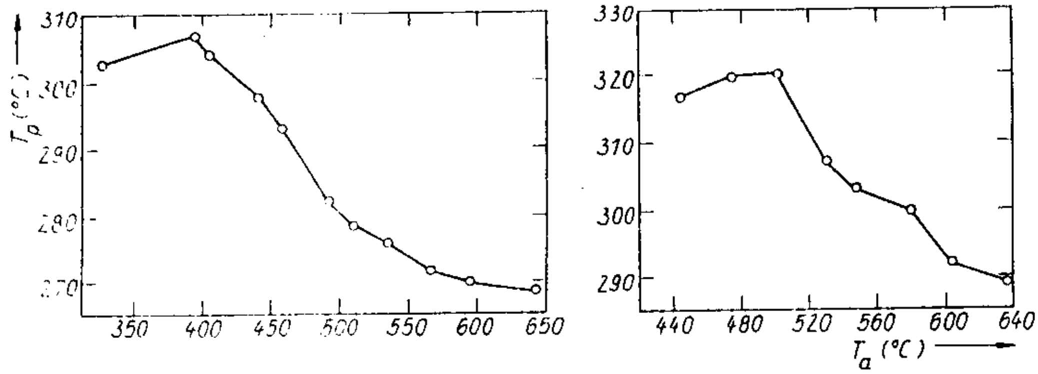


Fig. 4 Variation of T_p with T_a for a) 0.5 mm and b) 1.0 mm sheet specimen

Kẻ, Fig. 4

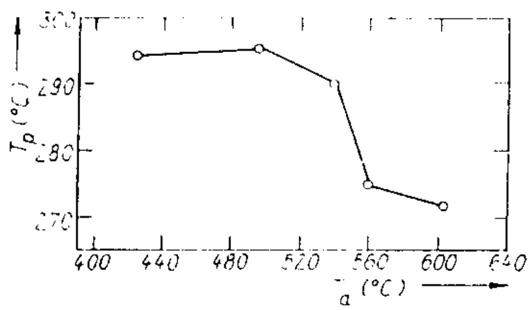


Fig. 5 Variation of T_p with T_a for specimen with diameter of 0.7 mm

Kẻ. Fig.5.

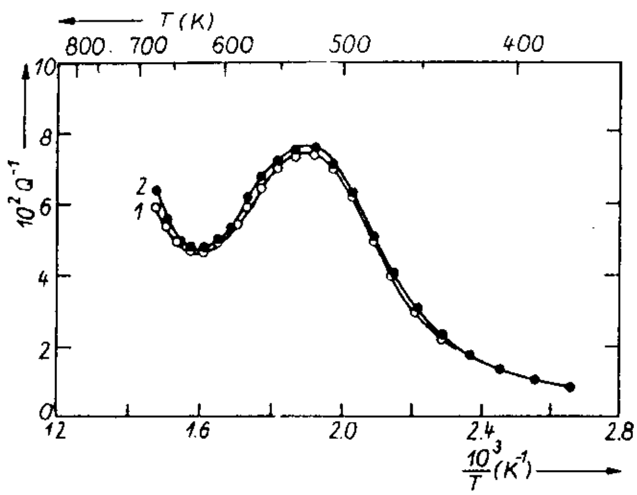
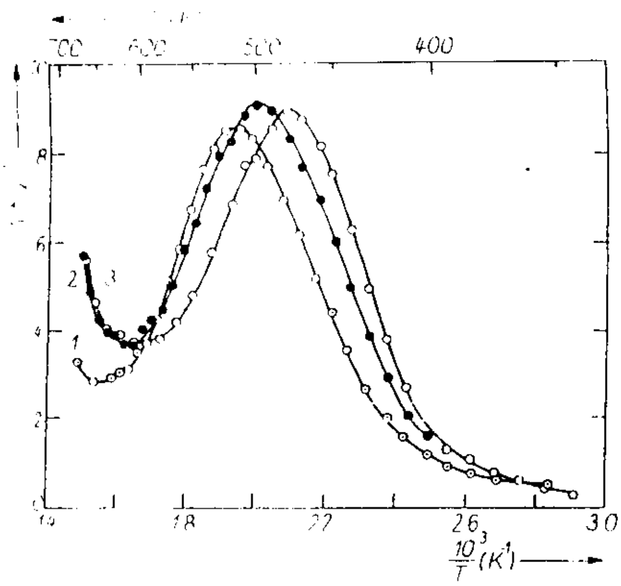


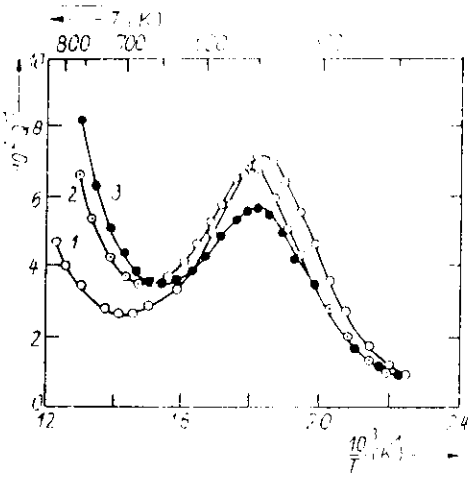
Fig. 6 Effect of surface Al_2O_3 layer on the macrocrystalline peak in 99.999% aluminium; (1) without surface Al_2O_3 layer, (2) with a thick Al_2O_3 surface layer

Kẻ, Fig.6.



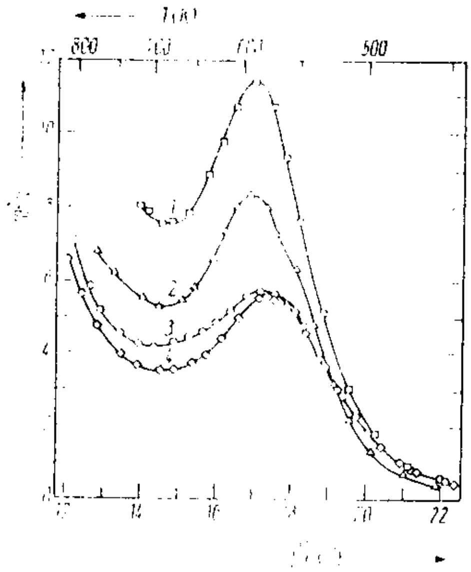
Kč. Fig. 7.

Fig. 7. Effect of annealing temperature on the position of the macrocrystalline peak of 99.999% aluminium (Japanese-made): (1) 450 °C anneal for 2 h, $GS \approx 1.1$ mm, (2) 500 °C anneal for 2 h, $GS \approx 2.2$ mm, (3) 600 °C anneal for 2 h, $GS \approx 2.5$ mm; diameter of specimen 1 mm



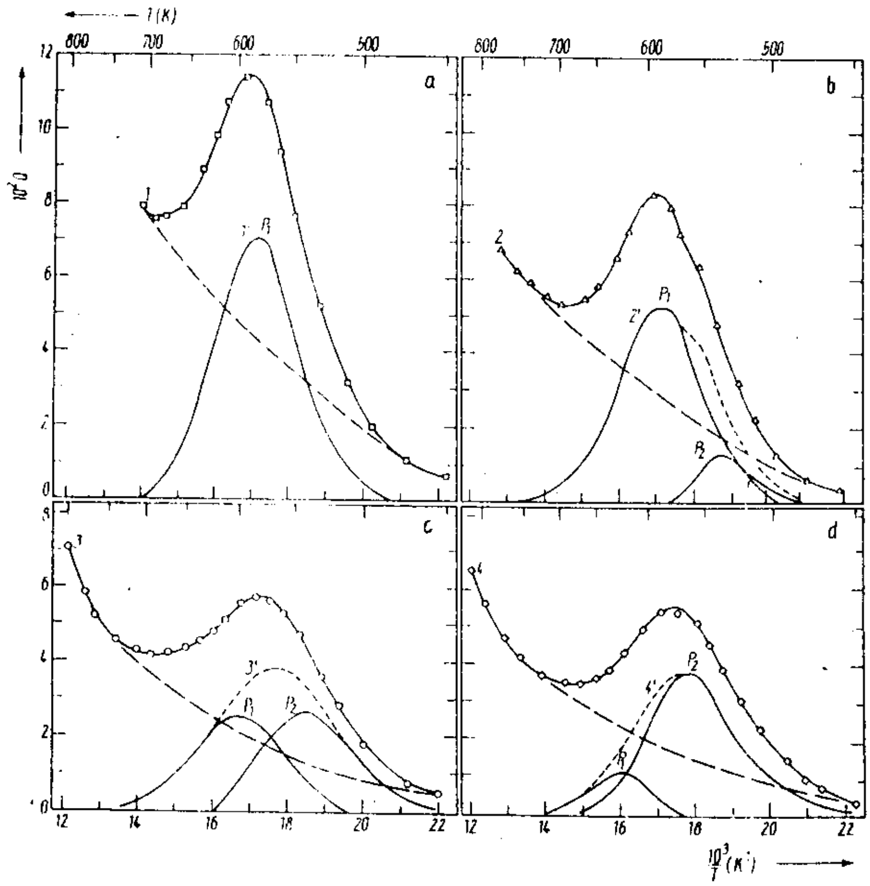
Kč. Fig. 8.

Fig. 8. Effect of creep deformation on the macrocrystalline peak of 99.999% aluminium: (1) without creep deformation, (2) 1.28% creep deformation at 495 °C, (3) 2.83% creep deformation at 495 °C



Ké, Fig. 9.

Fig. 9 Internal friction curves of cold-rolled sheet specimen annealed at various temperatures. Curves 1 to 4 correspond to annealing temperatures of 445, 580, 610, and 640 °C



Kê, Fig. 10

Fig. 10 Resolution of the internal friction curve a) 1, b) 2, c) 3, and d) 4 of Fig. 9

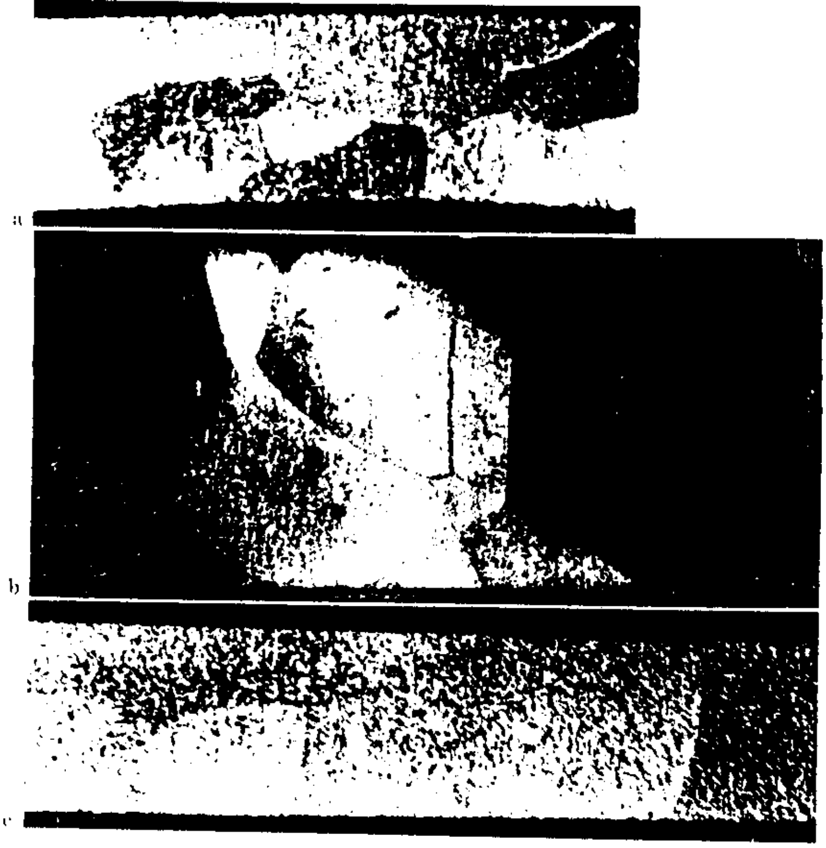
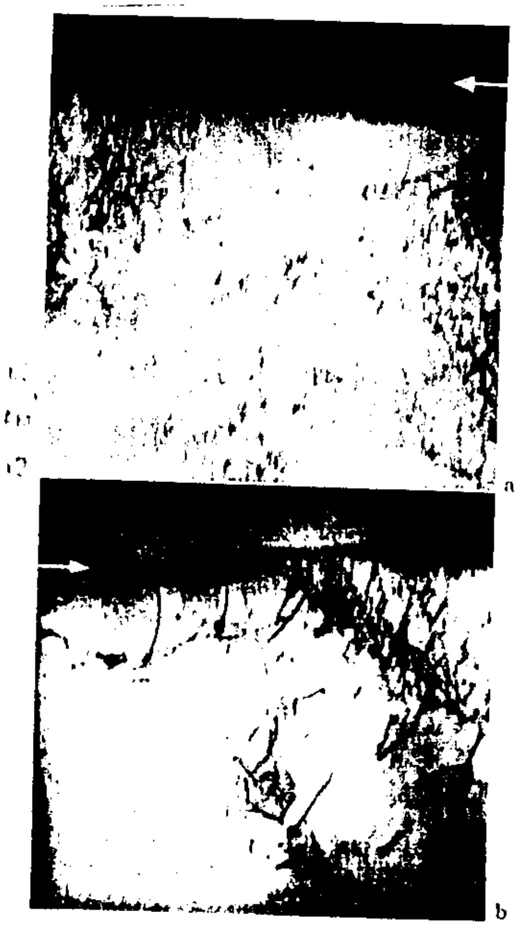


Fig. 11 Metallographs taken on the surface of cold-rolled sheet specimen annealed at a) 530 °C (× 25), b) 610 °C (× 45), c) 640 °C (× 25)



Kè Fig.12

Fig. 12 a) Cell structures near bamboo boundary (450 °C annealing) (15000 ×). b) Sharpened cell structures near bamboo boundary (600 °C annealing) (20000 ×). The bamboo boundary is shown by the arrow

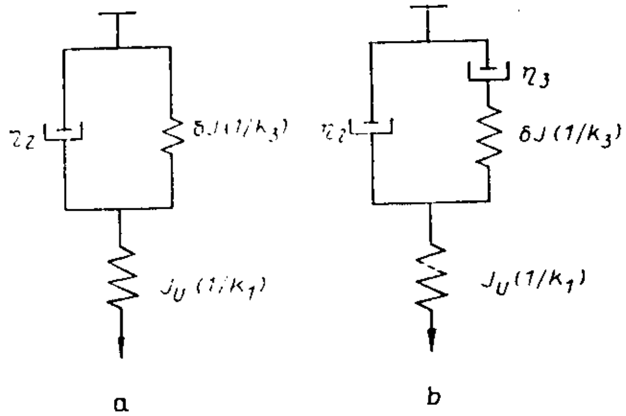


Fig.13 Mechanical models describing the relaxation behavior of grain boundaries. a) Fine-grained grain boundaries, b) bamboo grain boundaries

Kê, FRR.

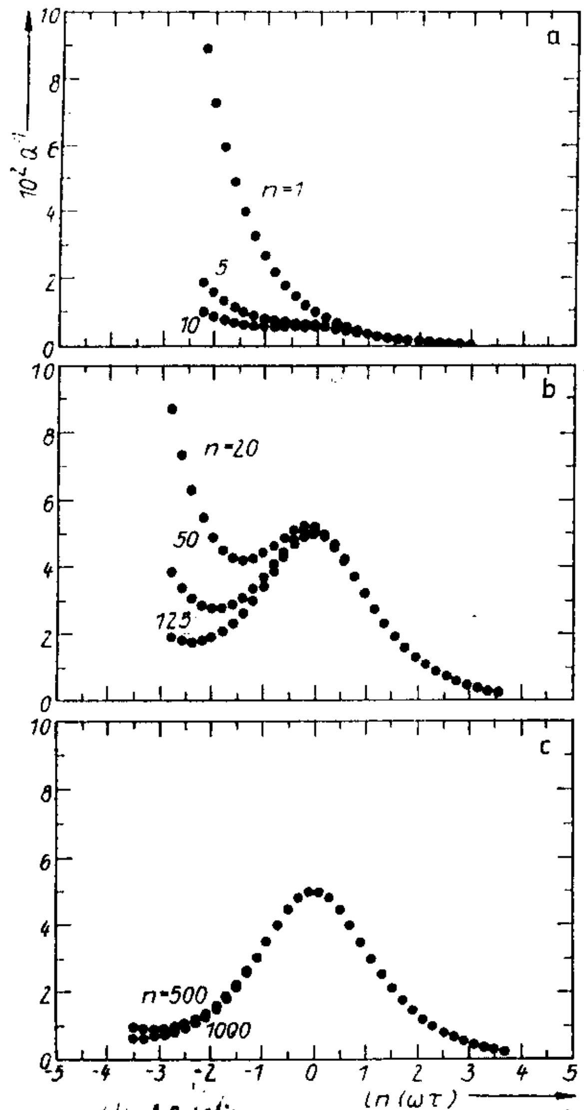


Fig.14 Internal friction curves showing tan φ as function of ln ωτ for various values of $n \equiv k_1/k_3$ (cf Fig.13 for the meaning of k_1, k_3 and η_2, η_3)

Kê Fig.19.

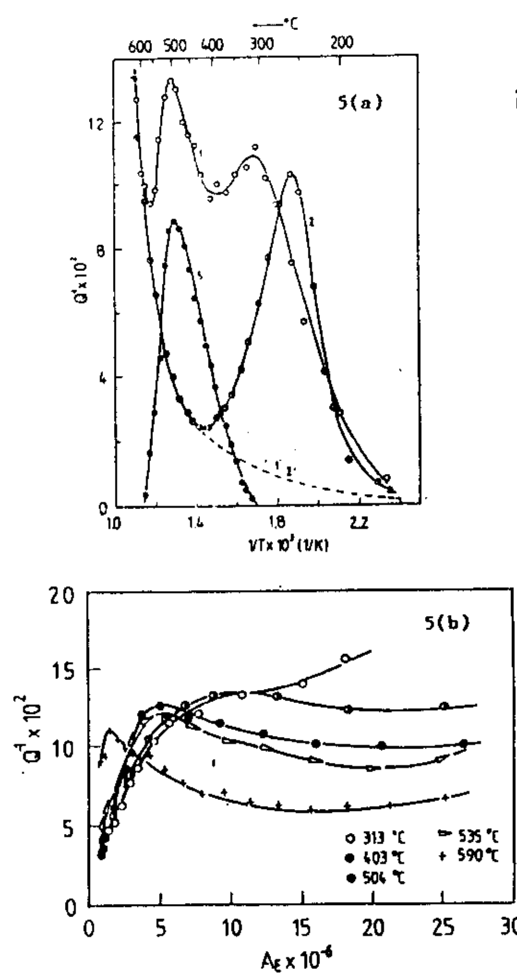


Fig.15(a)HT peak (curves 5)for 650℃ quenching.
(b)Amplitude peaks at temperatures indicated.

Kê, Fig.R