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Ultra-Micro Hardness Testing and Microscopic Deformation of Polycrystalline Aluminum

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The evaluation of microscopic inhomogeneity of polycrystalline aluminum is performed by measuring the hardness in respective grains. The recently developed ultra-micro hardness tester is used and the effects of the test pattern, the indentation load and the indenting velocity are examined. Then, the relationship between the increase in the hardness caused by the work hardening and the deformation of respective grains are statistically investigated.

The hardness testing mode in which the initial load is applied before the onset of measurement gives more stable results than the testing mode without the initial load. The test condition with the indentation load of 9.8mN and the indentation velocity of 0.2 μ m/sec seems to be optimum and gives the least dispersion of the measured values in grains. It is shown that the hardness values of respective grains in polycrystalline aluminum as well as their dispersion increase with the applied plastic strain. Discussion is made on the microscopic deformation behavior of polycrystalline aluminum.

1. INTRODUCTION

Hardness testing is one of the fundamental methods to examine the characteristics of materials(1). Hardness is the measure of the resistance of materials mainly related to the plastic deformation. The hardness testing is convenient and stable in measuring the characteristics of the specimen. There is another advantage that the local material characteristic can be estimated with the hardness measurement.

Recently, the ultra-micro hardness testing equipment has been developed which makes it possible to measure the hardness with very small load. The size of the indentation is also very small when the load becomes minute, and the error becomes large in the measurement with human eyes. Therefore, in the ultra-micro hardness testing machine, the hardness is usually obtained by measuring the depth of the indentation.

Many of the metallic materials used for industry is polycrystalline and their deformation behavior is microscopically heterogeneous. That is, when evaluating the material characteristic of polycrystalline metals with the hardness, the local heterogeneity such as grains must be considered. In order to clarify the basic characteristics of the ultra-micro hardness testing machine, pure aluminum is adopted in the present study, and the inhomogeneous deformation of polycrystalline metal is evaluated quantitatively. The influence of the testing mode, the indentation load and the indentation velocity are studied at first. Then, the hardness values of local area in respective grains and their dispersion are obtained. The hardness values in respective grains are also measured before and after tensile deformation, and the change in local hardness and its dispersion with the plastic deformation are examined.

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2. EXPERIMENTAL PROCEDURES

2. 1 Specimen and Tensile Test

The material used in the present study is pure aluminum (99.5wt%) for industrial use. The shape and the size of the specimen are shown in Fig.1. The specimens were annealed at 470℃ for 18hr in a box furnace. Next, the specimens were electropolished to remove the surface layer.

The tensile testing equipment with the capacity of 1960 N loaded by the screw and hand was used. Four areas of $2 \times 2\,mm^2$ were marked on the specimen and used for the area of the hardness measurements. The length between the marked lines and the grain boundaries were measured and the logarithmic strain of the specimen as well as the grains was calculated.

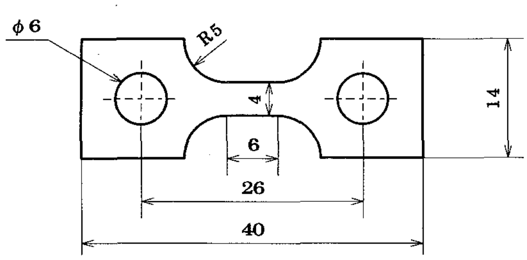


Fig. 1 Shape and dimensions of specimen (1 mm thickness)

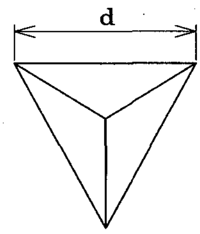


Fig. 2 Shape and size of pit of ultra-micro hardness indenter

2. 2 Hardness Testing

The hardness is measured with the ultra-micro hardness testing machine (Type MZT-4 produced by Akashi Ltd). The triangular pyramid indenter shown in Fig. 2 is used in the machine, where the angle between the surface and the axis of the indenter is 68 degree. The indentation load is applied with the electromagnetic force and the indenter is pushed into the sample. The indentation depth is measured with the displacement gauge of the capacitance type.

First, the indentation hardness HUT is obtained from the indentation load and the surface area of the pit. The surface area is calculated from the measured indentation depth and the hardness value is obtained from

$$HUT = 2.972\ F / h^2 \ . \qquad (1)$$

F : Indentation load (mN), h : Indentation depth (μ m)

Because the hardness is obtained from the load and the depth at the process of pressing the indenter into the specimen surface, it is said that elastic deformation in addition to plastic deformation affects the HUT hardness.

On the other hand, the triangular pyramid hardness HMT is given by measuring the diagonal length of the pit of the indentation after unloading, similarly to the well known micro-Vickers hardness testing method.

$$HMT = 218.23\ F / d^2 \qquad (2)$$

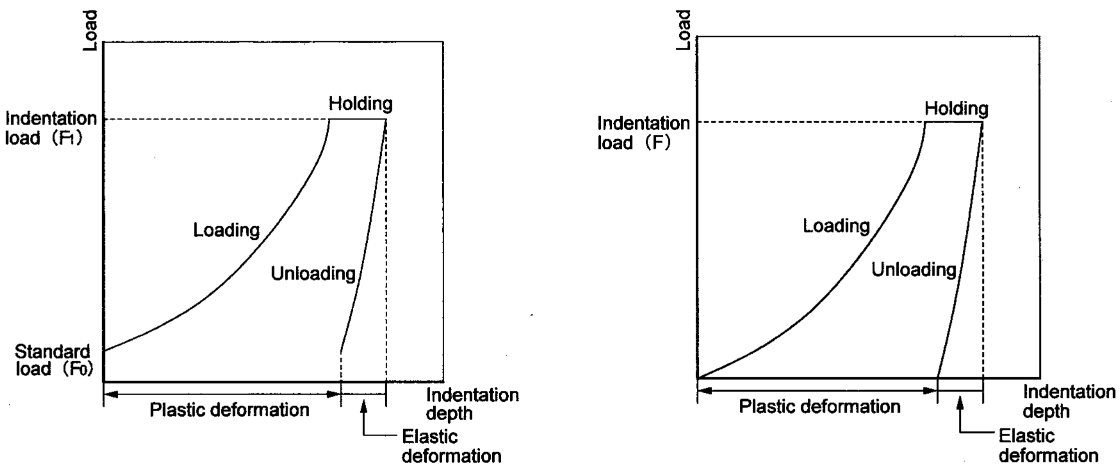
F : Indentation load (mN), d : Diagonal length of indentation pit (μ m)

The HMT hardness is considered to express the material characteristic of the plastic deformation, for the hardness obtained by measuring the size of the pit.

2. 3 Testing Modes of Hardness Measurement

Two main testing modes of hardness, that is, Test mode A and B are employed in the present study and the results are compared.

In Test mode A shown schematically in Fig. 3 (a), two kinds of load are set at the beginning, that is, the initial standard load and the final load. The standard load is applied first and that point is considered as the zero depth of the indentation. Then, the load is increased up to the maximum load. The maximum load is hold for a certain period and then the unloading process continues until the load decreases to the standard load.



(a) Test mode A (b) Test mode B

Fig. 3 Indentation load-depth curve

The hardness value of Test A is given from Eq. (1) as follows.

$$HUT = 2.972\,(\sqrt{F_1} - \sqrt{F_0})^2 / h^2 \qquad (3)$$

F_1 : Maximum load (mN), F_0 : Standard load (mN),

H : Indentation depth (μ m)

The application of the standard load is expected to reduce the influences of the surface roughness and the surface hardened layer.

Fig. 4 shows an example of the measured indentation load-depth curve. The patterns of the indentation load-depth curve are similar even if the examination load is changed. The depth increases continuously to the maximum loading point and becomes deeper during the holding time. At the time of unloading, the depth decreases due to the recovery of elastic deformation.

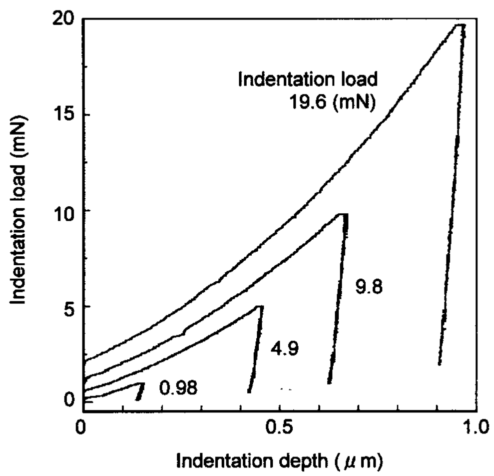


Fig. 4 Example of indentation load-depth curve
(Test mode A , Indentation velocity : 0.1 μ m/sec,)

Meanwhile, in Test mode B shown schematically in Fig. 3 (b), the load is analyzed like Test A, but there is no initial standard load at the beginning of the load application. Therefore, at the zero displacement point, the indenter first contacts the specimen surface.

The hardness value of Test B is also calculated from Eq. (1) and Eq. (2). In the present study, both Test modes A and B are performed at first where the condition of measurements are set to be almost equal and discussion is made on the method having higher reliability based on the measured data. Then, the obtained optimum condition is applied to measure the hardness before and after tensile deformation.

2.4 Problems on Various Hardness Measurements

In the present study, hardness was measured for the grains with relatively clear boundary line and lying near the center of the specimen. When measuring the hardness, the measuring points were separated by about 5 times of the pit width in order to prevent the change of the hardness value by the interference between the pits. Similarly, the measuring points were left the grain boundary by about 5 times of the pit width to avoid the influence of grain boundary on the hardness value.

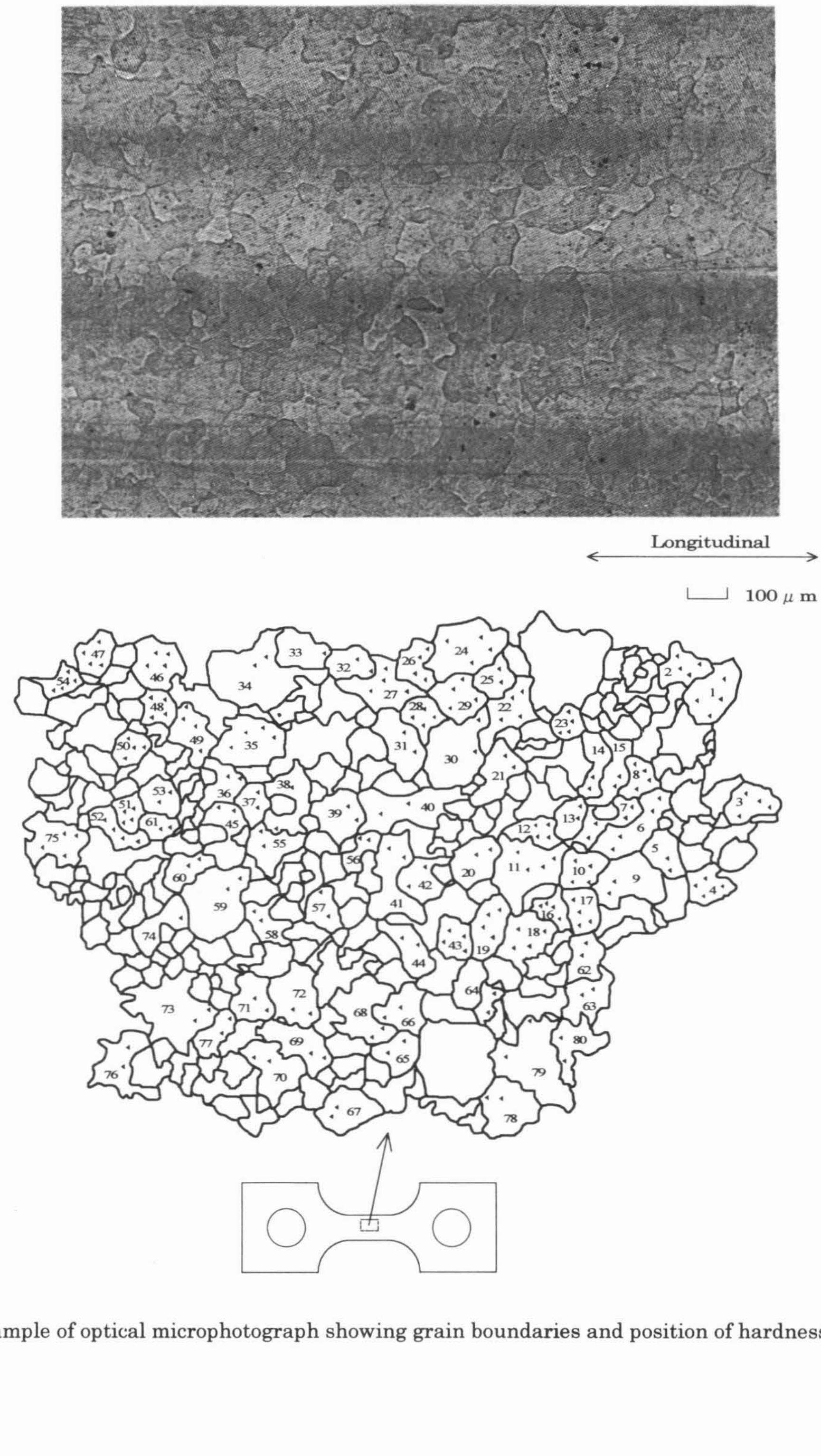


Fig. 5 Example of optical microphotograph showing grain boundaries and position of hardness measurement

2. 4. 1 Comparison of Test mode A and B

At first, the hardness values and its dispersion obtained by Test mode A and Test mode B, respectively with and without the initial standard load, are compared. As for Test A, the initial standard load was chosen as 1/10 of the maximum testing load. Furthermore, the indentation velocity is constant for Test A, while the loading rate is constant for Test B. In order to compare the results obtained from Test A and B, Test B is done at first and then the indentation velocity for Test A was determined from the expected indentation depth and the loading time given as follows.

(Loading time) = (Indentation load) / (Indentation speed) . (4)

The loading rate of Test B was chosen so that the loading time becomes in between 5-60sec.

2. 4. 2 Optimum condition for hardness measurement in grain

The purpose of this experiment is to find the optimum indentation load and the indentation velocity from the experiments with different indentation load and indentation velocity in grains. First, after the best indentation velocity is determined for Test mode A, the hardness values in one grain is measured with various testing load and the most suitable load is obtained. The adopted indentation loads were 0.98, 4.9, 9.8 and 19.6 mN, where the holding time and the unloading time were both 10 sec. For each measuring condition of the indentation load and the indentation velocity, the hardness was measured for three points in every five grains. Then, for the obtained optimum load, measurements with various indentation velocities were performed in order to find the optimum velocity. Namely, the indentation velocities of 0.02, 0.06, 0.1 and 0.2 μm /sec were tested.

2. 5. 3 Change in hardness before and after tensile deformation

An example of the optical microphotograph, the map of the grain boundaries and the position of measurement is shown in Fig. 5. Fig. 5 is composed of 35 photographs and made in the personal computer. Based on the grain boundary map, almost 80 grains were chosen which have enough size to measure the hardness. Two points in respective grains were measured before and after the tensile deformation. After the tensile deformation, however, slip lines appeared and the micro-hardness measurement becomes difficult in several grains, and finally the change in hardness was obtained for about 30 grains.

3. EXPERIMENTAL RESULTS AND DISCUSSION

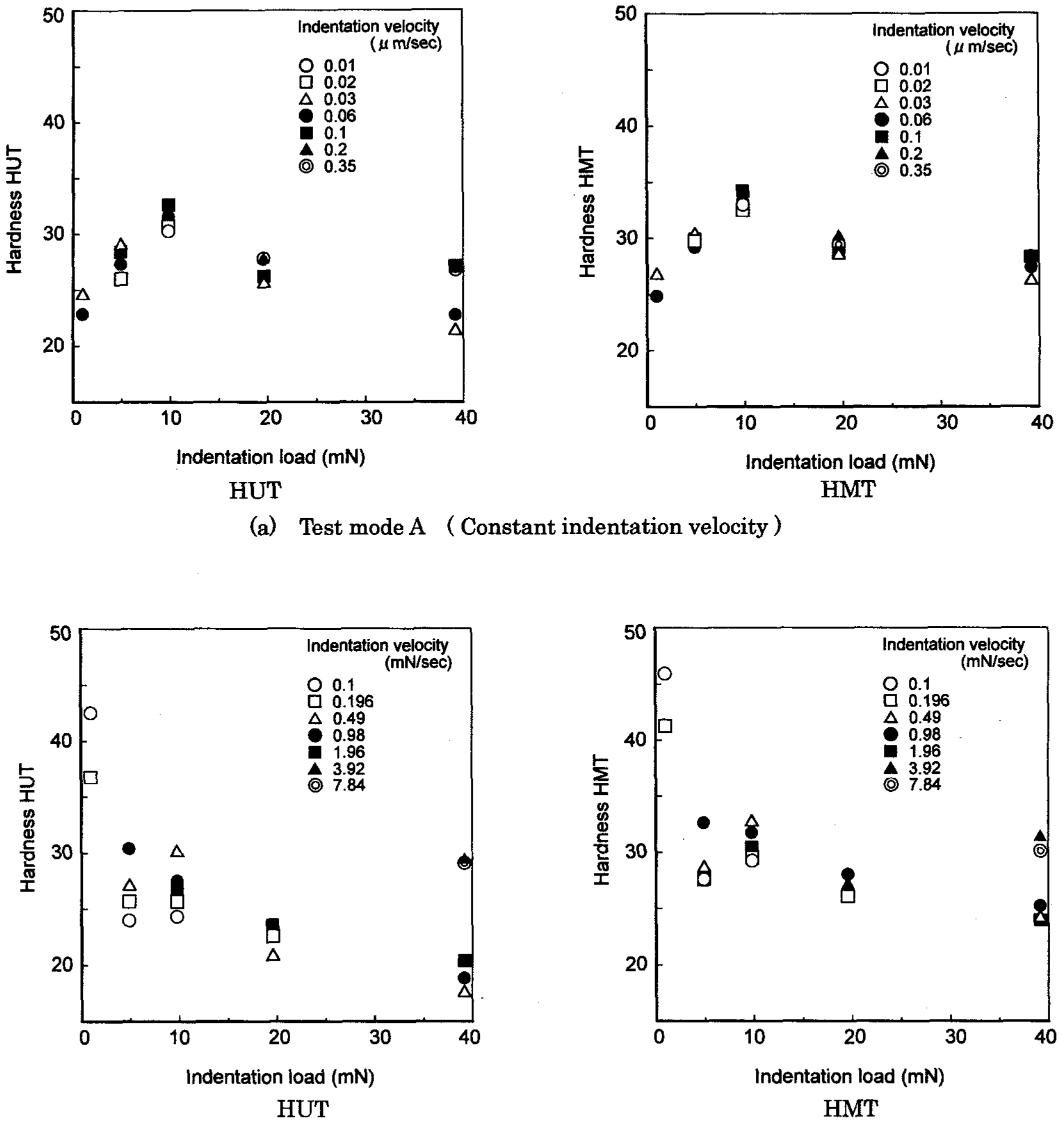
3. 1 Hardness Obtained from Test Mode A and B

In order to examine the difference in hardness values and the dispersion of the data obtained by Test mode A with the standard load as well as by Test mode B without it, hardness was measured for random five points in several grains and the averaged values were compared. Fig. 6 shows relation between the indentation load and the hardness values. The value of the indentation velocity in Test mode A was set to become similar to that in Test mode B.

The obtained HMT value (the triangular pyramid hardness value) is larger than the HUT value (the indentation hardness value) for all the testing conditions of with Test A and B. The HUT hardness corresponds to the hardness both for the elastic and the plastic deformation, while the HMT hardness corresponds to the hardness for plastic deformation alone. Hence, the elastic recovery occurs in the HMT and the indentation depth decreases, which is considered to result in the high hardness value.

In Test B, the value of the hardness at 0.98 mN becomes large as compared with the other testing loads. As Test B does not have the initial standard load and the indentation depth are about 0.27 μm, then the measured hardness value is affected by some surface layer. Fig. 7 shows the relation between the hardness and the indentation velocity. It is seen from Fig. 6 and 7 that the hardness value decreases with the increasing load both for Test mode A and B.

The influence of the indentation velocity on the hardness is relatively small. When the dispersions of the hardness values for Test A and Test B are compared in Figs. 6 and 7, the dispersion is smaller in Test A than that in Test B. It is considered that this is because the standard load is applied in Test A which removes the influences of the surface roughness and the surface work-hardened layer. Therefore, Test A seems to be more reliable, which is chosen in the following experiment.



(a) Test mode A (Constant indentation velocity)

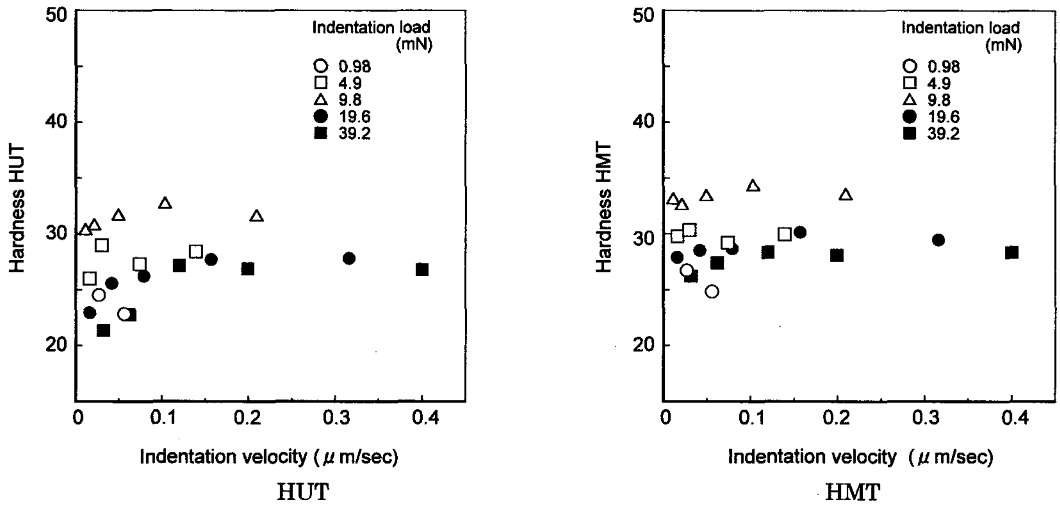
(b) Test mode B (Constant loading velocity)

Fig. 6 Relation between hardness and testing load

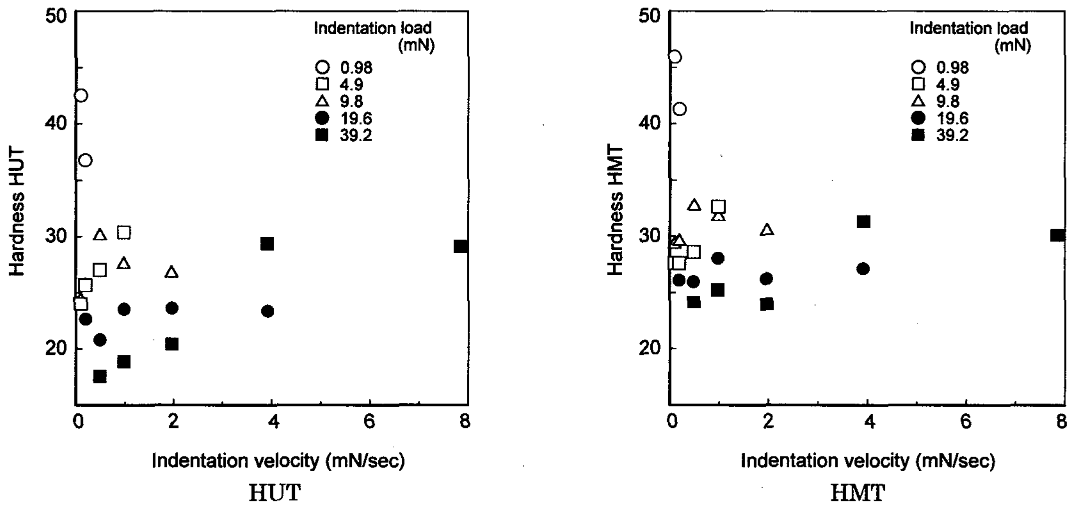
3. 2 Optimum Condition for Hardness Measurement in Grains

In the experiment mentioned in Section 3.1, the influence of the difference of grains was included. Here, Test A was performed for three points in respective grains changing the testing load and the indentation velocity. From the averaged values for the three measured values, the optimum testing load and indentation speed were determined.

First, the smallest indentation speed 0.1 μ m/sec with good dispersion of the measured hardness values is employed in Fig. 7 (a) of Test A, and the optimum testing load is tested. The relation between the hardness and the testing load for respective grains is shown in Fig. 8, where the results seem to be approximately constant for the load larger than 9.8 mN. The value of hardness becomes rather high at the load 0.98 mN compared with the other testing load. This may be resulted from the surface layer, for the indentation depth is as small as about 0.17 μ m.



(a) Test mode A (Constant indenting velocity)



(b) Test mode B (Constant loading rate)

Fig. 7 Relation between hardness and indenting velocity

Figure 9 shows the relation of the difference between the maximum and the minimum hardness values versus the indentation load in respective grains. It is seen from Figs. 8 and 9 that when the indentation load is less than 9.8 mN, the dispersion of the hardness value in respective grains is large and the hardness value itself is relatively large. Therefore, it is concluded that the optimum indentation load for the present problem is 9.8 mN or 19.6 mN. Considering the size of the pits, however, it may be said that the optimum indentation load is 9.8 mN.

Next, in order to examine whether the indentation speed 0.1 μ m/sec is optimum for the optimal indentation load 9.8 mN, the indentation velocity is changed. Fig. 10 shows the relation between the hardness and the indentation velocity in grains. Fig. 11 shows the difference between the maximum and the minimum hardness value versus the indentation velocity.

It is seen from Figs. 10 and 11 that the dispersion of the hardness values in respective grains as well as that among grains is smaller for the velocity 0.2 μ m/sec than that for the velocity 0.1 μ m/sec, except for the grain 2 in which the dispersion of the data is relatively large. Then, the optimum indentation velocity is determined as 0.2 μ m/sec in the following measurements.

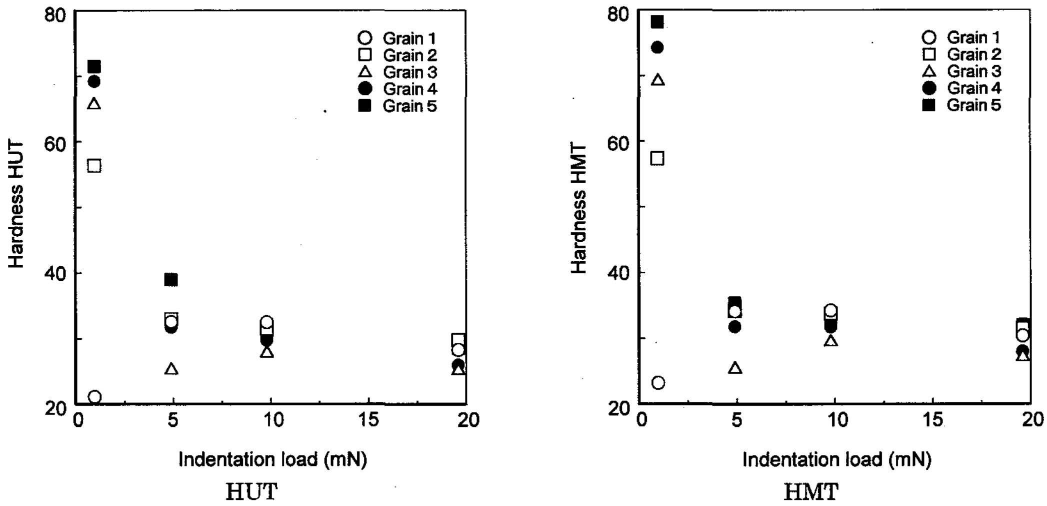


Fig. 8 The relation between hardness and indentation load in respective grains (Indentation velocity : 0.1 μ m/sec)

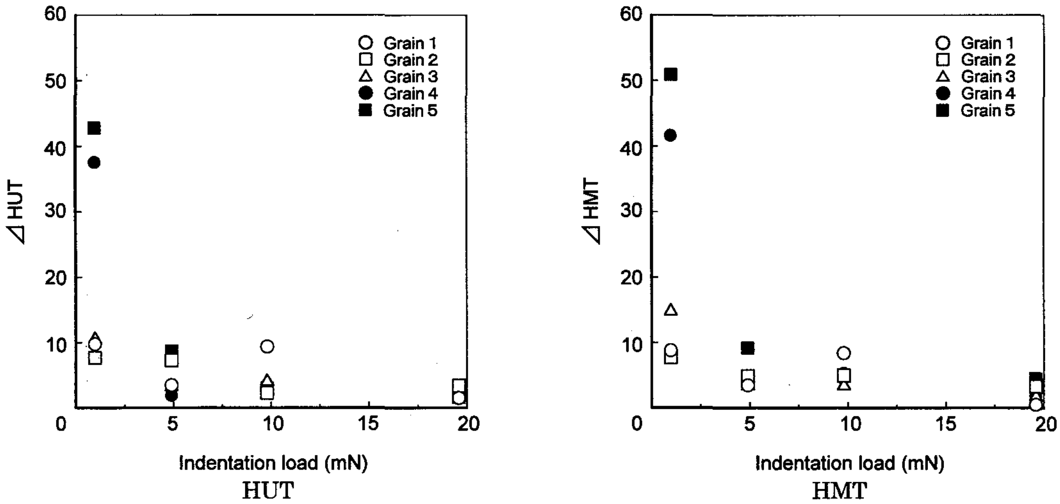


Fig. 9 Relation of difference between maximum and minimum hardness values versus indentation load in respective grains (Indentation velocity : 0.1 μ m/sec)

3. 3 Dispersion of Hardness in Respective Grains

Next, hardness measurement was done under the condition of the indentation load 9.8mN and the indentation velocity of 0.2 μ m/sec, which was confirmed to be most reliable. The dispersion of hardness values in each grain and for different grains were obtained from the hardness obtained from ten measurements in respective grains. Fig. 12 shows the measured hardness and its average in respective grains. Some errors may be included in the HMT measurement of pits, for their size is as small as 6-9 μ m and hence there may be some difference among individuals in measuring the size.

3. 4 Change in Hardness Before and After Tensile Deformation

Here, the change of the hardness in respective grains before and after tensile deformation are examined. Fig. 13 shows the distribution of hardness HUT in respective grains. It is seen that the averaged hardness value increases from 30.9 to 42.7 with the tensile straining. Meanwhile, the value standard deviation increases form 2.98 to 4.71. It may be noted that the variation coefficient, that is, the ratio of the standard deviations to the averaged hardness is 0.10 and 0.11, respectively, before and after deformation. Namely, the ratio is almost unchanged with the tensile deformation.

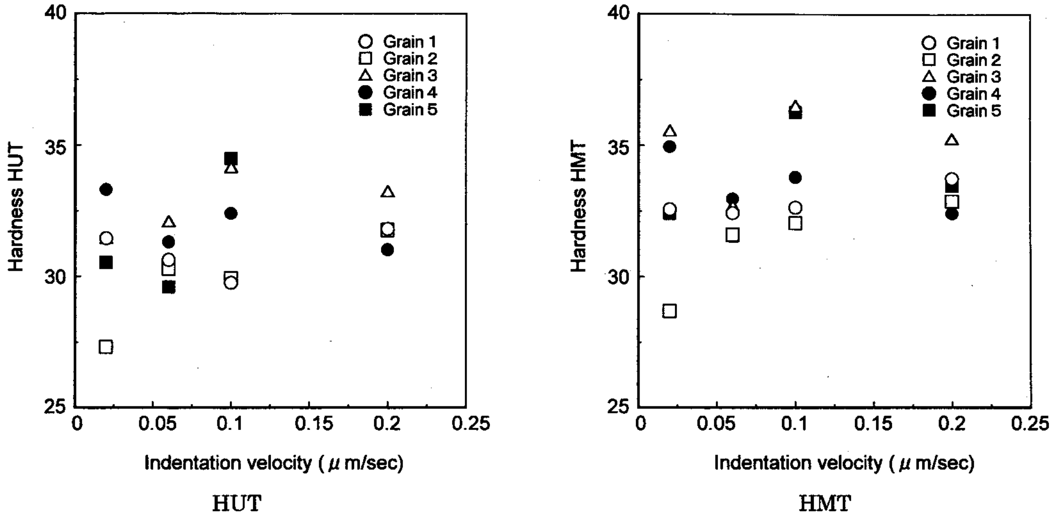


Fig. 10 Relation between hardness and indentation velocity in respective grains
(Indenting loads : 9.8 mN)

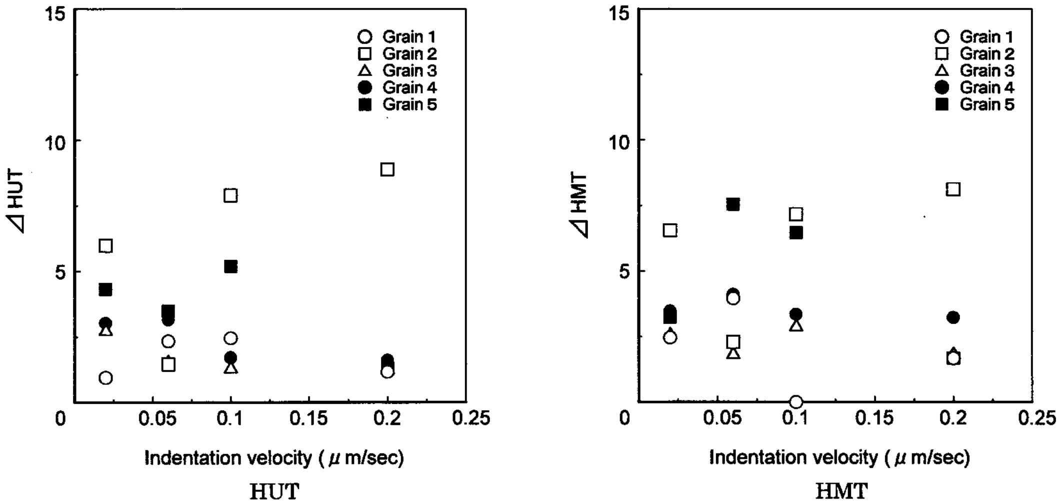


Fig. 11 Relation between increase in hardness and indentation velocity for respective grains
(Indenting load : 9.8 mN)

Figure 14 shows the relation between the hardness increment before and after the deformation and the hardness before deformation of respective grains. Although the dispersion in respective grains is comparatively large, it is seen that hardness increment is large for the grains having smaller value of the initial hardness. This trend was also reported by Kamikawa et al [(2)] using another type of the ultra-micro hardness testing machine. Namely, it may be said that those grains with small initial hardness are comparatively much work-hardened after deformation.

Figure 15 shows the distribution of the grain strain in the axial loading direction as well as in the perpendicular direction. The strain is obtained from the maximum width of the grain boundary in each direction measured on the grain map and represented by the logarithmic strain. Although there is a certain dispersion of the grain strain, their averaged value is 0.139, which is close to the macroscopic strain $\varepsilon = 0.10$ of the specimen.

Figure 16 shows the relation between the hardness after deformation and the strain of grains. Clear correlation, however, is not observed in Fig. 16 and further investigation on this point seems to be necessary.

Figure 17 shows schematic diagram of hardness (flow stress) versus strain relation, estimated from the result shown in Fig. 14. Fig. 17 explains the situation that the initially soft grains increase their hardness much during plastic deformation.

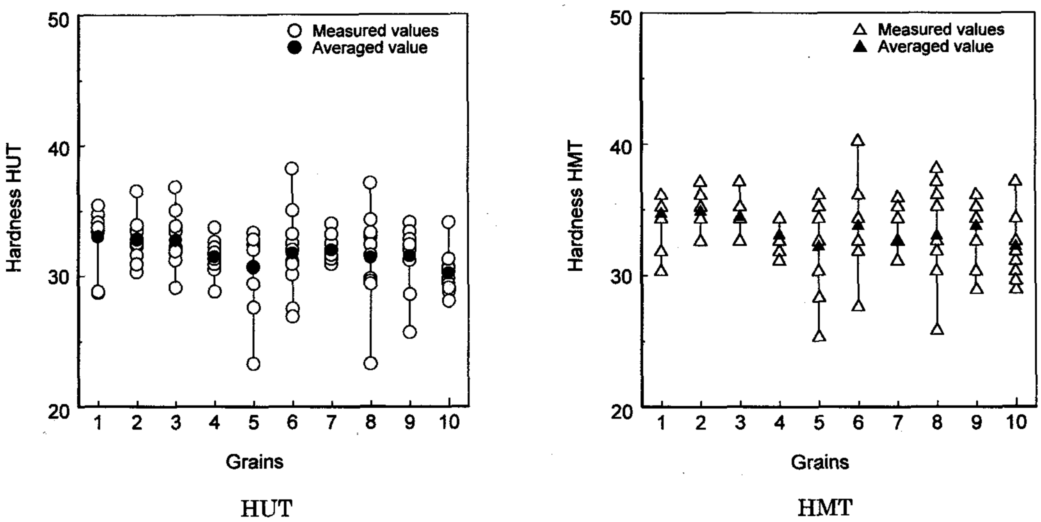


Fig. 12 Measured hardness value s and averaged value in respective grains

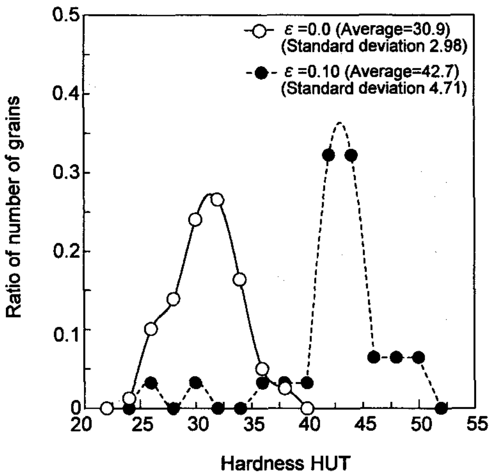


Fig. 13 Distribution of hardness value in respective grains before and after tensile deformation

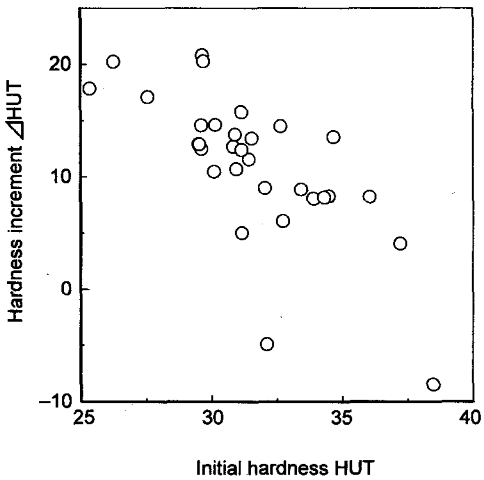


Fig. 14 Relation between hardness value before deformation and hardness increment after deformation

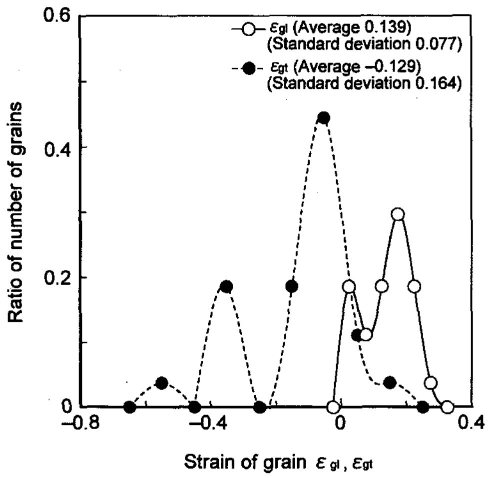


Fig. 15 Distribution of strain for respective grains

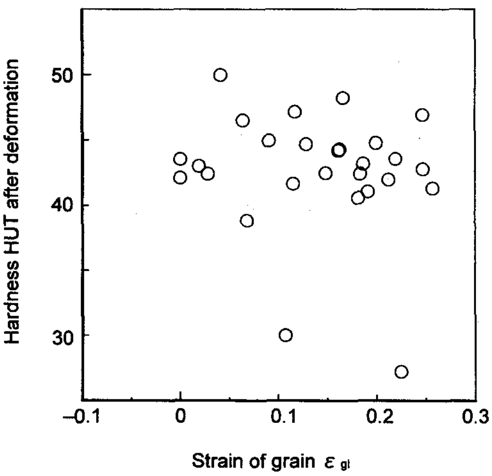


Fig. 16 Relation between hardness after deformation and axial strain of grains

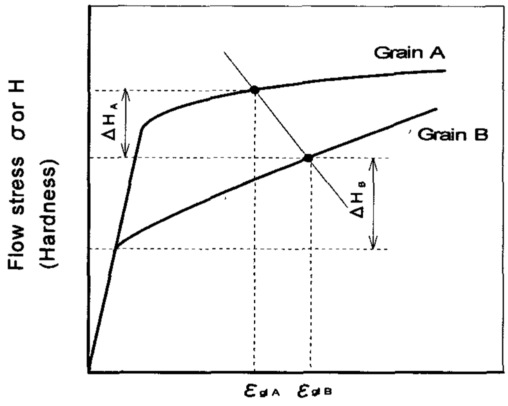


Fig. 17 Schematic diagram of hardness (flow stress) versus strain of polycrystalline metal

4. CONCLUSIONS

In order to study the microscopic plastic deformation of polycrystalline metal using ultra-micro hardness testing apparatus, the hardness testing were performed for pure aluminum specimens. The local hardness and its dispersion in and among grains were measured, changing the testing conditions such as the testing patterns, the indentation load and the indentation velocity. The hardness change in respective grains was measured before and after the uniaxial tensile deformation. The deformation of respective grains was also measured. The main results are as follows.

(1) The dispersion of the hardness values is smaller for the hardness measurement with the small initial load than the measurement without it, which indicates the former method is more reliable than the latter. This may be due to the influence of the surface roughness and the work-hardening layer produced during specimen preparation.

(2) The measured hardness shows some dependence on the load for the range between 0.98mN-39.2mN under the indentation velocity of 0.1 μ m/sec.

(3) The condition of the load 9.8 mN and indentation speed 0.2 μ m/sec gives the lowest dispersion in grains.

(4) The hardness value in respective grains becomes large with the applied strain, owing to the work-hardening of the grains. The dispersion of the hardness also increases with the applied strain. This fact shows that the non-uniform deformation in and among grains increases with the applied strain. The ratio of the dispersion to the mean hardness value, however, is almost unchanged.

(5) The smaller the hardness value before deformation is, the larger the degree of work-hardening after plastic deformation. This shows that the degree of work-hardening after the deformation is large in those grains favorably oriented for the deformation under the applied stress,.

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