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General paper

Lüders Deformation and Strain Aging of [001] Oriented Fe-30%Cr Alloy Single Crystals

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Abstract: Peculiar deformation accompanied by Lüders band having 46% Lüders strain was observed in the [001] oriented Fe-30%Cr alloy single crystal. Specimen was again deformed after aging at 473K for 1.8ks at the 23.5% strain. The first Lüders band can not move due to locking by aging and second Lüders band started from the opposite side of the chuck. The specimen having [001] tensile axis is thought to begin to slip ideally by four slip directions. In the present case, this specimen deformed by slip of [111] and $[11\bar{1}]$ directions. Thus, the slip system of Lüders band could be determined. From the surface observation of the side surface of the specimen, the propagation speed, width and the strain-rate in the Lüders band were determined. The mobile dislocation density in the Lüders band was also estimated to be about 2×10^8cm/cm^3. The effective and internal stresses were determined during the straining in order to analyze the mechanism of Lüders deformation.

Key words: *Plastic deformation, Fe-30%Cr single crystal, Lüders band, Strain aging*

1. INTRODUCTION

In ferritic steels having bcc structure, not so many systematic investigations have been performed on deformation and fatigue by employing orientation controlled single and bicrystals. This is because a ferritic steel has transformation points and it is difficult to make specially oriented single crystals by the Bridgeman method. However, in Fe-Cr alloy having higher than 13Cr, the α-single phase can be obtained and this makes possible the study of bcc single crystals by growing well orientation controlled single crystals.

Ferritic high chromium alloys are used as commercial steels due to low price, fairly well corrosion resistance and good weldability. Therefore, the present study was undertaken in order to clarify the strength and mechanism of deformation of commercial Fe-Cr ferritic stainless steels.

In has been reported by Kaneko [1] that Fe-30%Cr alloy single crystals having [001] tensile axis were fractured by cleavage at room temperature accompanied by twinning. However, after careful heat treatment avoiding impurity contamination, it was found to be possible to cause deformation of up to about 100% elongation at room temperature. This deformation proceeded by the Lüders band propagation just after beyond the yield point. It was found the peculiar deformation was accompanied by Lüders band having 46% Lüders strain. Then this sample was deformed after aging at 473K for 1.8ks, another Lüders band started and propagated from the opposite side of the chuck. From the series of deformation experiments, we report the observation of slip bands, the propagation speed of Lüders band front, width, the strain rate and the mobile dislocation density in the Lüders band.

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2. SPECIMEN

Fe-30%Cr alloy single crystal was made from polycrystalline rods by the Bridgman method. Chemical composition of the polycrystalline rods used is presented in Table 1. The carbon content is 28ppm. The specimens with shoulder gauge of 6mm in length, 0.5mm in thickness, 3mm in width were cut by spark cutter to be [001] oriented. The specimens were annealed at 1273K for 3.6ks and then quenched into a water and were electrolytically polished. Due to mechanical polishing and repeated electrolytical polishing, the final thickness was reduced to be 0.46mm.

Growing of single crystals were carried out in the Materials Science Laboratory of Department of Physics and Mechanics, Kyoto University.

Table 1. Chemical composition of material (mass%).

C	Si	Mn	P	S	Cu
0.0028	0.010	<0.005	0.0005	0.0019	<0.001
Ni	Cr	Al	O	N	Fe
<0.001	30.09	<0.001	0.0004	0.0158	Bal.

3. EXPERIMENTAL PROCEDURE

Tensile tests were carried out with an Instron type testing machine made by Shimadzu Co. Ltd. (AGS500B). Strain-rate change test was performed using 1:10 of strain-rate from $\dot{\varepsilon}_1=1.38\times10^{-3}$/s to $\dot{\varepsilon}_2=1.38\times10^{-2}$/s Test temperature is room temperature, otherwise noted. At first, the specimen was deformed by 23.5%. After aging at 473K for 1.8ks and then the specimen was again deformed until 37.6%. Also, high temperature deformations up to 548K were made.

4. METHOD OF ANALYSIS

4.1 Method of Measurements of the Propagation Speed, Width, Strain-Rate and the Density of Mobile Dislocation in the Lüders Band

Wijiler and Van Den Beukel [2] showed that denoting the strain $\Delta\varepsilon$, the width d, the propagating velocity V_B and the strain-rate $\dot{\varepsilon}_B$ of the Lüders band, and consequently the mobile dislocation density during the Lüders deformation can be determined by measuring the local strains of two points or one point of the specimen as a function of time.

Grip (cross head) speed $\dot{g}$ of tensile machine is given as follows [3].

$$\dot{g} = V_B \times \Delta\varepsilon . \quad (1)$$

$$\dot{g} = \dot{\varepsilon}_B \times d . \quad (2)$$

The following equations are deduced for the strain-rate of the Lüders front.

$$\dot{\varepsilon}_B = \rho_m \times b \times v_d , \quad (3)$$

where b and v_d is Burgers vector and the average velocity of the moving dislocations. Combining Eqs.(1), (2) and (3),

$$\rho_m = \frac{V_B}{v_d} \times \frac{\Delta\varepsilon}{b \times d} . \quad (4)$$

Now following Hahn [4], the value of V_B can be taken as the same order of magnitude as v_d, therefore we can write,

$$\rho_m = \frac{\Delta\varepsilon}{b \times d} . \quad (5)$$

In order to estimate ρ_m from Eq.(5), $\Delta\varepsilon$ and d have to be determined experimentally.

In the case of this experiment, let the gauge length of original sample is l_0, length of undeformed region is l_1 as shown in Fig. 1 and the time required for propagation of the Lüders band is t,

$$V_B = \frac{(l_0 - l_1)}{t} . \quad (6)$$

And the strain-rate $\dot{\varepsilon}_B$ of Lüders band can be written

$$\dot{\varepsilon}_B = \frac{\Delta\varepsilon}{\Delta t} , \quad (7)$$

where Δt is the time required for a Lüders band propagates through the width d of Lüders band.

Therefore, width d of Lüders band can be expressed by

$$d = V_B \times \Delta t . \quad (8)$$

Also, it becomes from Eq.(2),

$$d = \frac{\dot{g}}{\dot{\varepsilon}_B} . \quad (9)$$

4.2 Method of Determining of Effective Stress and Internal Stress during Deformation by Strain-Rate Change Test

Strain-rate sensitivity exponent m^* was given by $m^* = \Delta \ln \dot{\varepsilon} / \ln \sigma^*$, which is the ratio of change of effective stress σ^* when strain-rate $\dot{\varepsilon}$ was changed, the value of m^* was calculated by Eq.(10) by means of m-values obtained during deformation was extrapolated to the strain of starting plastic deformation $\varepsilon_{p=0}$.

$$m^* = \left(\frac{\Delta \ln \dot{\varepsilon}}{\Delta \ln \sigma^*} \right) = \left(\frac{\Delta \ln \dot{\varepsilon}}{\Delta \ln \sigma} \right)_{\varepsilon_{p=0}} = m_{\varepsilon_{p=0}} . \quad (10)$$

In the Fe-30mass.%Cr alloy single crystal, the m^* obtained by tensile test and strain-rate change during deformation. Consequently, the increase of alternating quantity $\Delta\sigma$ of maximum tensile stress σ_{max} by strain-rate change from $\dot{\varepsilon}_1$ to $\dot{\varepsilon}_2$ before and after, is used to calculate the effective stress σ^* during deformation by Eq.(11) [6],

$$\sigma^* = \Delta\sigma / \{ (\dot{\varepsilon}_2 / \dot{\varepsilon}_1)^{1/m^*} - 1 \} . \quad (11)$$

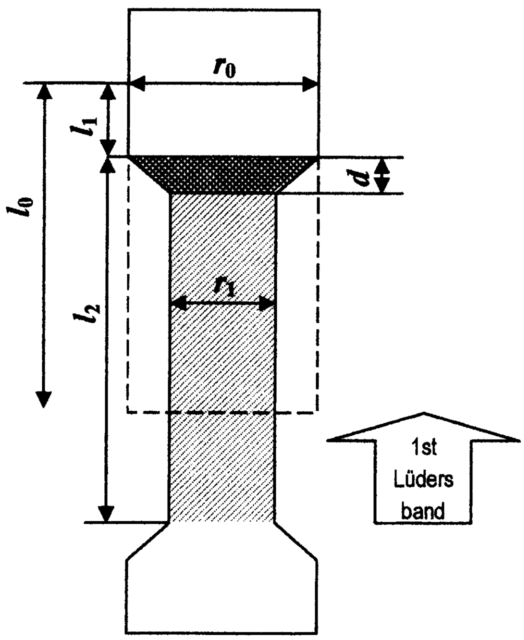

Fig. 1. Schematic drawing of the Lüders band.

This method of calculation of m^* is based on the concept that m^*-value is not dependent on strain and mode of deformation. Such independency has been reported in deformed pure Fe crystals.

According to Seeger [7], a flow stress σ consists of a thermal component (σ^*: effective stress) and an athermal component (σ_i: internal stress) as shown in Eq.(12), therefore, σ_i and σ^* was calculated from this equation.

$$\sigma = \sigma_i + \sigma^*(T, \dot{\varepsilon}) \cdot \quad (12)$$

5. RESULTS AND DISCUSSION

Stress-strain curve of the [001] Fe-30%Cr single crystal was shown in Fig. 2. The yield point appeared after aging. This is due to the fact that the first Lüders band which occurred at first deformation didn't proceed and propagate, however the second Lüders band started from the opposite side of the chuck and propagated. Figure 3 shows the appearance of the specimen deformed by 23.5%. As described later, left half of the specimen was deformed by 46% which corresponds the Lüders strain.

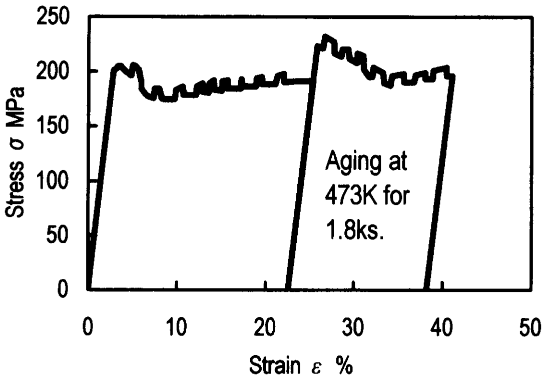

Fig. 2. Stress-strain curve of [001] Fe-30%Cr single crystal.

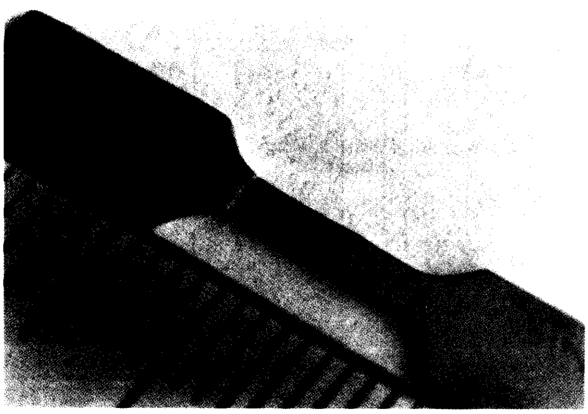

Fig. 3. Surface observation of [001] Fe-30%Cr single crystal deformed by 23.5% strain at room temperature.

The specimen was annealed at 473K for 1.8ks and then deformed again by 37.6%. The appearance of the specimen is shown in Fig. 4. It can be seen that the second Lüders band has started from the opposite side of the chuck. The Lüders strain of second one was found to be 50%.

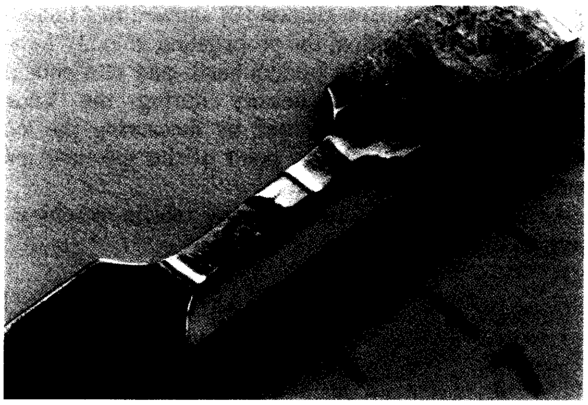

Fig. 4. Surface observation of [001] Fe-30%Cr single crystal deformed by 37.6% strain after aging at 473K for 1.8ks at 23.5% strain.

5.1. On the First Lüders Band

5.1.1. Slip

The specimen having [001]tensile axis is thought to begin to slip ideally by four slip directions, [111] $[\bar{1}11]$ $[1\bar{1}1]$ $[11\bar{1}]$, spontaneously, however, the slip steps of slip lines with $[\bar{1}11]$ and $[1\bar{1}1]$directions can not be observed because slip directions are parallel to the top surface. On the other hand, slip lines of [111] and $[11\bar{1}]$ can not appear on the side surface since slip directions are parallel to the side surface as shown in Fig. 5, only the slip of [111] and $[11\bar{1}]$ directions could be analyzed. Also it cannot distinguish these two slip directions from the observation of top surface, because slip with [111] and $[11\bar{1}]$ directions are equivalent. However, after the tensile deformation, it was determined that the slip lines on the side surface are slip with $[11\bar{1}]$ direction and a following analysis was made as slip with $[11\bar{1}]$ direction.

The slip direction was analyzed using the photograph of Fig. 5 which was taken from the side surface of specimen. The strain in the region where the first Lüders band have passed could be determined as follows by using the measured values of r_0=0.46mm and r_1=0.25 mm, determined by direct measurements of thickness.

$$\Delta\varepsilon_1 = (r_0 - r_1)/r_0 = 0.46 = 46\% \, .$$

Slip lines observed on the side surface were analyzed as they belong to the $(11\bar{2})$ $[11\bar{1}]$ slip system. From the Fig. 5 (right) it is found that the angle between [001] and [111] is 55.0°. If we suppose that the propagated area of Lüders band was deformed by 46%, the angle between slip line and the tensile axis must rotate from 55.0° to 33.2°. The measured value was 30.0° and found to be almost in agreement with the calculated value.

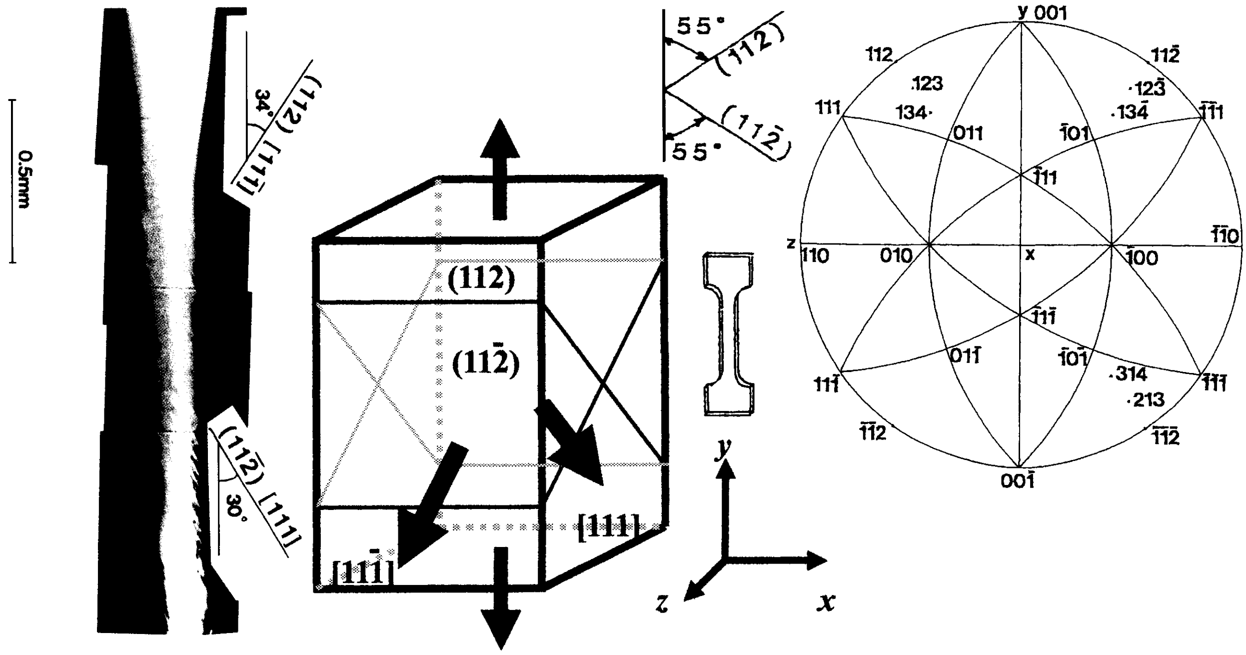

Fig. 5. Side surface observation of [001] Fe-30%Cr single crystal at 23.5% strain.

Next, we will examine whether the real elongation of the specimen can be explained by the deformation of Lüders band or not. The real elongation of the specimen was found to be 23.5% and the gauge length was l_0=6.0mm. The total strain can be given as

$$\varepsilon = \frac{\Delta l}{l_0} = \frac{(l_0 - l_1)}{l_0} \times \Delta\varepsilon_1, \qquad (13)$$

where Δl is the specimen length supposed to be elongated by Lüders deformation, l_0 is the original gauge length of the specimen, l_1 is the length of undeformed region and $\Delta\varepsilon_1$ is the Lüders band strain.

At first, we will consider the region (l_2-d), where the complete deformed region of Lüders band. As it is seen that l_1=2.4mm l_0=6.0mm and d=0.9mm, then the total strain is calculated as 21% from the Eq. (13). On the other hand, if we consider the region of Lüders front l_2, similarly, the total elongation was calculated to be 28%. Thus the average value is 24.5% which is almost in agreement with the real elongation 23.5%. This fact is considered that the this specimen deformed by an ideal Lüders band deformation.

5.1.2. On the propagation of Lüders band

At first, the lattice parameter a was calculated from the data by Preston [8] which is a=2.860+0.000186 ×x(Å), where at% of Cr is x. For the Fe-30mass%Cr, a is given by

$$a = 2.860 + 0.000186 \times 31.52$$

$$= 2.86\,\text{Å} = 2.86\times10^{-8}\text{cm}$$

Next, we calculate the mobile dislocation density ρ_m. As the Burger's vector of bcc crystal is b=(a/2)<111>, b is given as

$$b = \frac{a}{2} < 111 > = \left(\frac{\sqrt{3}}{2}\right) \times 2.87 = 2.48\times10^{-8}\,cm\text{'}.$$

Thus, if $\Delta\varepsilon$ is the strain in the first Lüders band and d is the width of the band, ρ_m would be

$$\rho_m = \Delta\varepsilon/(b\times d) = 0.46/(2.48\times0.09\times10^{-8})$$

$$= 2.1\times10^{8}(\text{cm/cm}^3).$$

In this case, the time required for the propagation of the first Lüders band was 183.6s. Therefore, the propagation speed of the first Lüders band V_{B1} is found to be

$$V_{B1} = (l_0 - l_1)/t = 3.6/183.6 = 0.0196\text{mm/sec}$$

$$= 2.0\times10^{-3}\text{cm/s}.$$

Here, we will consider the concept proposed by Hahn [4] that the propagation speed of Lüders band is nearly equal to the mean moving velocity of the dislocations. Now, the edge dislocation velocity in the Fe-3%Si crystals was measured by Stain and Low [9], which is at 298K about 10^{-3}cm/s at the stress 147MPa and 1cm/s at 196MPa. In the present investigation, the sample of Fe-30%Cr crystal, the dislocation velocity is 2.0×10^{-3}cm/s in the stress range from about 170 to 200MPa, this value is found in the range from 10^{-3} to 1cm/s and is almost in agreement with the values of Hahn's concept.

From the above result, Eq. (1) $\dot{g} = V_B \times \Delta\varepsilon$ and Eq. (2) $\dot{g} = \dot{\varepsilon}_B \times d$ will be examined. Here, the $\dot{g}$ is grip speed (Cross head speed), and the $\dot{\varepsilon}_B$ is strain rate of Lüders band. Propagation velocity of first Lüders band is V_{B1}=2.0×10^{-3} cm/s and width is d_1=0.9mm, therefore, the time t_1 required for propagation of the first Lüders band is

$$t_1 = d_1 / V_{B1} = 0.09/2.0\times10^{-3} = 45.0s.$$

The strain rate of Lüders band $\dot{\varepsilon}_{B1}$ is calculated by using t_1

$$\dot{\varepsilon}_{B1} = \Delta\varepsilon_1/t_1 = 0.46/45.0 = 0.0102/s.$$

From these values, $\dot{g}$ is obtained from Eqs. (1) and (2) as

Eq. (1)
$$\dot{g} = \dot{\varepsilon}_{B1}\times d$$
$$= 0.0102/s\times 0.9mm = 0.0092mm/s.$$
$$= 0.55mm/\text{min}.$$

Eq. (2)
$$\dot{g} = V_{B1}\times\Delta\varepsilon_1$$
$$= 2.0\times10^{-3}cm/s\times0.46 = 0.92\times10^{-3}\,cm/s.$$
$$= 0.55mm/\text{min}.$$

The actually used cross head speed of testing machine, namely grip speed is 0.5mm/min., which is almost in agreement with the value obtained above.

5.2 On the Second Lüders Band

The same analysis was made on the second Lüders band. Figure 6 showed the schematic drawing of side of the specimen surface after second tensile testing.

5.2.1 Slip

In order to determine the values of r_0 and r_2, the direct measurements of thickness were made and found to be r_0=0.46mm and r_2=0.23mm. Therefore the second Lüders band strain, $\Delta\varepsilon_2$ is

$$\Delta\varepsilon_2 = (r_0 - r_2)/r_0 = 0.50 = 50\%.$$

Also similar to first Lüders band, slip lines observed on the side surface were analyzed as they belong to the $(11\bar{2})$ [111] slip system.

Since the region of Lüders deformation deformed by 50%, the angle between tensile axis and slip line would be changed from 55.0° to 30.6°. Since observed angle is 30.0°, it is almost in good agreement (Fig. 7).

The real elongation of the specimen was found to be 37.6% and gauge length was l_0=6.0mm. The total elongation is expressed as

$$\varepsilon = \frac{\Delta l}{l_0} = \frac{l_0 - \left(l_3 + \frac{l_2}{1+\Delta\varepsilon_2}\right)}{l_0}\times\Delta\varepsilon_1 + \frac{l_0 - \left(l_3 + \frac{l_1}{1+\Delta\varepsilon_1}\right)}{l_0}\times\Delta\varepsilon_2 \quad (14)$$

where, l_1=5.0mm, l_2=1.7mm, the length of undeformed

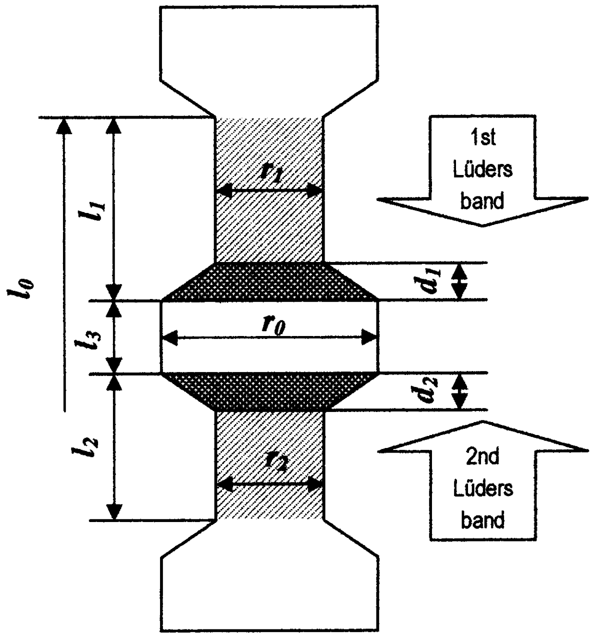

Fig. 6. Schematic drawing of the Lüders band of [001] Fe-30%Cr single crystal at 37.6% strain.

part l_3=1.6mm and l_0=6.0mm, d_1=0.9mm, d_2=0.7mm as shown in Fig. 6. At first, if we take the region (l_1-d_1) and (l_2-d_2) where deformed region of Lüders band, into account in Eq.(14), total elongation is calculated to be 33.2%. One the other hand, taking the region of Lüders front l_1 and l_2, into account, the total elongation was calculated to be 41.9%. If we take the average value of 37.6%, which agrees with measured value of 37.6%, thus, this specimen is considered to have been deformed by an ideal Lüders band deformation.

5.2.2 On the propagation of second Lüders band

Since the strain of the region of propagated second Lüders band was found to be $\Delta\varepsilon_2$=50%, the density of mobile dislocation was 2.02×10^8(cm/cm^3). The time required for propagation of the Lüders band t is 108s and the propagation speed of completely deformed region of Lüders band V_{B2} is

$$V_{B2}=\{l_0\times(1+\varepsilon_1)-(l_1+l_3)\}/t$$

$$=0.8/108=0.0075\text{mm/s}=7.5\times10^{-4}\text{cm/s}.$$

Therefore, the speed is fairly low compared with the propagation speed V_{B1}=2.0×10^{-3}cm/s of first Lüders band.

The time required for the front of second Lüders band propagates through the width d of Lüders band, t_2 is

$$t_2 = d_2 / V_{B2} = 0.07/7.5\times10^{-4} = 93.3s.$$

The strain rate of Lüders band calculated from t_2 is

$$\dot{\varepsilon}_{B2} = \Delta\varepsilon_2/t_2 = 0.50/93.3 = 0.00536/s.,$$

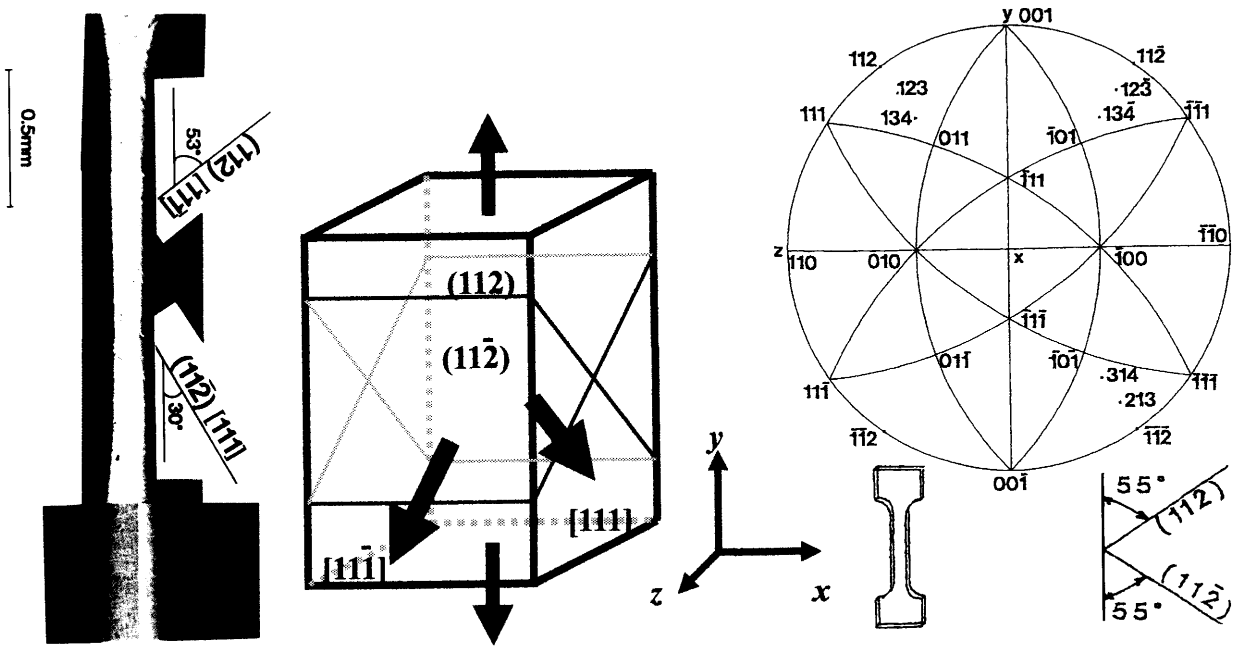

Fig. 7. Side surface observation of [001] Fe-30%Cr single crystal at 37.6% strain.

and this is half of $\dot{\varepsilon}_{B1}$ of first Lüders band.

5.3 On the *m*-Value, Internal Stress and Effective Stress

Figures 8 and 9 are shown the relation between *m* value and strain, obtained for [001] oriented crystals. It is seen from the figure that the *m*-value increases with strain. By extrapolating this curve to plastic strain 0, the m^*-value at 293K was obtained as m^*[001]=32.0. In the case at 548K, m^*[001]=15.0 was obtained. This value decreases with increasing deformation temperature, for example, the value of Fe-3%Si single crystal was reported to be 35.0 at 113K, 5.9 at 473K by Zarubova et al. [10].

Zarubova et al. [10] have shown that the moving of the group of parallel screw dislocation plays an important role not only in the beginning of deformation in which individual slip lines occur, but also do the same in the later stage of deformation, because the value is consistent with that obtained from the moving velocity of the screw dislocations at the front of a isolated slip line for this orientation in Fe-3%Si single crystal.

In this study, since the [001] oriented specimen was deformed by an ideal Lüders deformation, it will be expected that *m*-value which obtained from the measurement of propagation speed of the front of the Lüders band is consistent with the *m*-value obtained in this study.

Calculated results of internal stress σ_i and effective

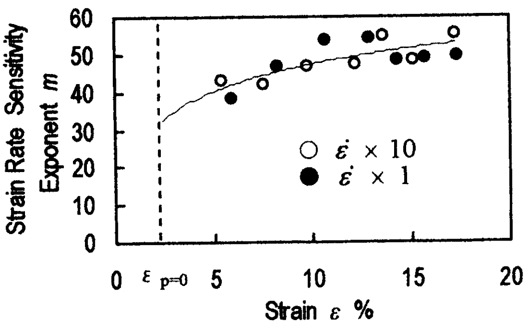

Fig. 8. Strain-rate sensitivity of a Fe-30%Cr single crystal measured at various strains at 293K. The open circles were data obtained during an increase in strain rate of a factor of ten (○), and the closed circles are points obtained during a decrease in strain rate (●). $\varepsilon_{p=0}$ indicates plastic strain 0.

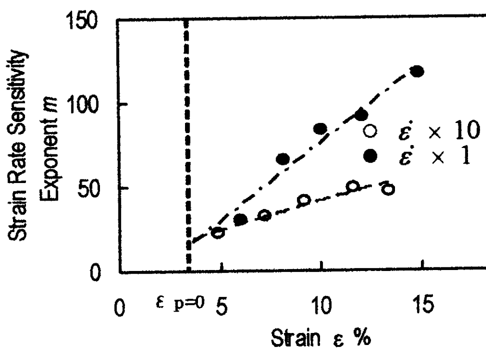

Fig. 9. Strain-rate sensitivity of a Fe-30%Cr single crystal measured at various strains at 548K. $\varepsilon_{p=0}$ indicates plastic strain 0.

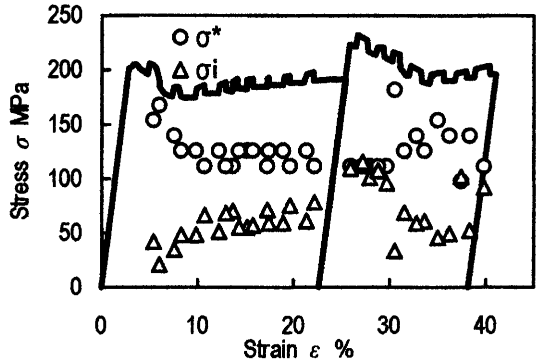

Fig.10. Stress-strain curve of [001] Fe-30%Cr single crystal σ^* and σ_i indicate effective stress and internal stress respectively. Specimen was aged at 473K for 1.8ks after stretching by 23.5%.

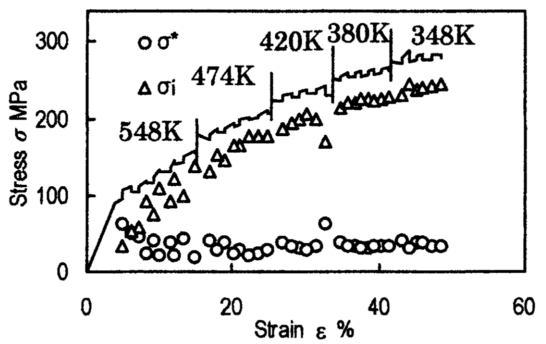

Fig. 11. Stress-strain curve of [001] Fe-30%Cr single crystal at various temperature. σ^* and σ_i indicate effective stress and internal stress respectively.

stress σ^* from this m^*-value were shown Figs 10 and 11. At room temperature deformation, σ^* is larger than σ_i and it was found that the σ_i is 30~40% of total applied stress and the σ^* is 70~60%. At high temperature deformation (548K), value of internal stress σ_i nearly occupies all of applied stress, showing that the effective stress is approaching to 0. The ratio of σ^* and σ_i is determined by where the deformation temperature T situated as shown in Fig. 12. It is shown in Fig. 12 that $\dot{\gamma}_1$ and $\dot{\gamma}_2$ are shear strain-rates and τ^* and τ_μ show the thermal (short range) and athermal (long range) components of the applied shear stress. Deforming at high temperature, the thermal component decreases to 0 at T_0, ie, at high temperature of T_0 at which all the deformation will be proceeded by an internal shear stress τ_i. As are seen in Figs 10 and 11 generally σ_i increases and σ^* slightly decreases or keeps constant with proceeding deformation. This fact means that the number of free mobile dislocations decreases with proceeding deformation. Increasing of σ_i is

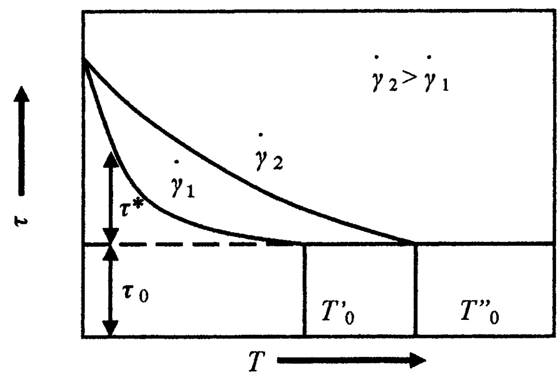

Fig. 12. Variation of stress with temperature and strain rate, showing thermal and athermal stress components [11].

considered that dislocations form cell walls and make strongly stabilized barriers. Generally, dislocations can overcome the short range barriers easily due to the rapid multiplication of free dislocations just after yielding occurs. In this study, σ^* is greatly decreases after the yielding point as shown in Fig. 10. In addition, Fig. 10 shows result that specimen again deformed after aged at 473K for 1.8ks after stretching, and it was found that σ^* increased. It is considered that many free dislocations were formed by unlocking during yielding because the first Lüders band was locked by the aging and second Lüders band took place from the opposite side of the chuck.

6. CONCLUSIONS

The results obtained from the room temperature deformation of [001] oriented Fe-30%Cr alloy single crystal are summarized as follows.

(1) The deformation was proceeded by the Lüders band propagation and this Lüders band has characteristic of having 46% Lüders strain.

(2) From the analysis of slip line and stereographic projection, it was confirmed that the preferred activation of the two slip systems among the quadruple slip systems of the [001] orientation.

(3) Deforming the specimen after aging, the first Lüders band was suppressed to move and the second Lüders band proceeded and propagated form the opposite side of the chuck where the Lüders band occur easily. The Lüders strain of the second band was 50%.

(4) Characteristics of the first Lüders band are that when the strain rate is 1.38×10^{-3}/s, the propagation speed is 2.0×10^{-3} (cm/s), width of band is 0.9mm, the strain-rate in the Lüders band is 1.02×10^{-2}(/s) and the mobile dislocation density in the Lüders band is obtained to be 2.1×10^{8}(cm/cm^3).

(5) Characteristics of the second Lüders band are that when the strain rate is 1.38×10^{-3}/s, the propagation speed is 7.5×10^{-4}(cm/s), width of band is 0.7mm, the strain rate

in the Lüders band is 5.36×10^{-3}(/s) and the mobile dislocation density in the Lüders band is obtained to be 2.02×10^{8}(cm/cm^{3}).

(6) During the deformation at room temperature, σ_i increases and σ^* decreases with proceeding of deformation. When the sample was deformed again after aging at 473K, the flow stress increased with showing yielding and σ^* increased. The first Lüders band can not move due to locking by aging and the second Lüders band occurred from the opposite side of the chuck. It is considered that the increasing of σ^* is due to the formation of many free dislocations by unlocking.

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