# Transmission Kossel Study of the Formation of (110) [001] Grains after an Intermediate Annealing in Grain Oriented Silicon Steel Containing a Small Amount of Mo

YUKIO INOKUTI, CHIZUKO MAEDA, and YO ITO

The transmission Kossel (TK) technique was applied in an attempt to elucidate the formation of (110) [001] grains in the recrystallized texture of high induction and low core loss grain oriented silicon steel containing a small amount of Mo after an intermediate annealing. Prior to the detailed TK examination, the etch pit technique was also used for seeking the representative areas of (110) [001] grains. The average matrix grain size of an intermediate annealed steel containing a small amount of Mo is slightly smaller than that with no Mo. The formation of (110) [001] grains in the vicinity of the steel surface is also strengthened due to the addition of Mo. The diameters of single (110) [001] grains are comparable to or slightly larger than those of other matrix grains, but those of the colonized (110) [001] grains occupy extremely large areas (about 40 pct at the maximum area). The areal fraction of (110) [001] grains of the intermediate annealed sheets containing a small amount of Mo is approximately 1 to 5 times as large as that with the conventional inhibitors such as MnSe and Sb. The colonies of (110) [001] grains formed during an intermediate annealing are compatible with the volume fraction inherited from small and well-defined (110) [001] areas with the sharpened TK patterns in the hot-rolled sheet containing a small amount of Mo. It is believed that the preferential formation of (110) [001] grains during an intermediate annealing is inherited by the structure memory from the original hot-rolled texture, and it is noticeably strengthened with the addition of Mo.

## I. INTRODUCTION

SINCE the oil crisis in 1973, energy saving has been strongly emphasized. The development of products with an ultra low core loss as well as a high magnetic induction has become an urgent necessity. At present, the loss evaluation system, in which a bonus is given for grain oriented silicon steel with an ultra low core loss, has been generally adopted in the United States and European countries.

The following methods[1] to make the core loss of grain oriented silicon steel as low as possible are generally considered: (1) the increase of silicon content in steel, (2) the decrease of the sheet thickness in products, (3) the promotion of purity in products, and (4) the preferential formation of secondary grains with small grain size, under which the formation of the celebrated (110) [001] secondary grains has been preserved. In products manufactured with methods (1) and (2), increasing the silicon content from 3 pct and decreasing the ordinary sheet thickness from 0.35 mm–0.30 mm to 0.23–0.20 mm markedly deteriorates the formation of (110) [001] secondary grains. Also, as the silicon content in grain oriented silicon steel increases, hot embrittlement becomes pronounced, and intergranular fracturing occurs during slab soaking or hot rolling. This serious problem spoils the surface quality of the final products. On the other hand, it is generally accepted that methods (3) and (4) will be at the maximum levels permitted by the present manufacturing processes. For example, with highly oriented products, the direction of easy magnetization parallel to the [001] axis deviates no more than 3 to 4 degrees[2] from the rolling direction, and in an attempt to obtain the celebrated (110) [001] secondary grains as small as possible while preserving high magnetic induction, it will be very difficult from a metallurgical aspect. The metallurgical review[2] for the preferential formation of the celebrated (110) [001] secondary grains has been published.

Recently, it has been found that with an increase of silicon content to about 3.4 pct a new silicon steel product containing a small amount of Mo makes it possible to obtain low core loss as well as high magnetic induction,[1,3] and can improve noticeably the surface quality of final products.[4,5]

Transmission Kossel (TK) technique,[6] which is capable of obtaining diffraction patterns from a point source of divergent monochromatic X-rays at subsurface of a specimen, is applied to measure precisely the orientation of small grains of about 5 to 20 $\mu$ in diameter. By use of this technique, it is possible to obtain detailed information of primary and secondary grains.[2]

In the detailed TK study[7,8] of hot-rolled steel containing a small amount of Mo, the frequency of generation and the areal fraction of the potential nuclei of (110) [001] secondary grains have been observed to be approximately three times as large as that of hot rolled Mo free steel. Therefore, it is believed that silicon steel containing a small amount of Mo has a lower core loss due to the development of small (110) [001] secondary grains. Although the preferential formation of small (110) [001] secondary grains due to the addition of Mo will be inherited by the structure memory*

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*The terminology of the structure memory[9] has been used as the sequence of the principal (110) [001] texture variation in the vicinity of the surface from the original hot-rolled steel; (110) [001] texture formation in the hot rolling→{111}⟨112⟩ in the first cold rolling→(110) [001] in the intermediate annealing→{111}⟨112⟩ in the second cold rolling→(110) [001] in as-decarburized and primary recrystallized annealing. Also, this terminology has been initially used by Gol'dshteyn *et al.*,[10] which is defined as repeated texture changes during cold rolling and annealing.

from the original hot-rolled steel, it is very important to study how the memory from the hot-rolled structure can be accomplished and how the (110) [001] primary grains are formed.

The objectives of the present work are to investigate the formation of (110) [001] grains after an intermediate annealing prior to the second stage of cold reduction in silicon steel containing a small amount of Mo with the aid of the TK technique and to compare the experimental results with those of steel containing no Mo.[9]

## II. SPECIMEN PREPARATION AND EXPERIMENTAL PROCEDURE

The materials used in the present investigation were intermediate annealed sheets of about 0.8 mm thickness with a chemical composition of 0.044 pct C, 3.35 pct Si, 0.07 pct Mn, 0.020 pct Se, 0.025 pct Sb, and 0.013 pct Mo. The method for producing silicon steel containing these elements[11] and the slab-soaking and hot rolling methods[12] have already been given. The important points in the production of silicon steel are to keep impurities as low as possible, to decompose and dissolve all the selenides during slab soaking, and to provide a fine dispersion of MnSe precipitates during hot-rolling. The first stage of cold reduction was done from hot-rolled sheets of 2.7 mm thickness to about 0.8 mm, and then intermediate annealing for 3 minutes at 950 °C was done with a continuous annealing furnace in the atmosphere of 50 pct $H_2$ and 50 pct $N_2$ mixed gas.

To elucidate the mechanism of preferential formation of the (110) [001] primary grains which can be inherited by the structure memory from the hot-rolled steel containing a small amount of Mo, specimens taken from a depth of 1/10 the sheet thickness under the steel surface and from the center of the sheet thickness were used in the TK examination and Schulz X-ray method. Thin specimens for TK examination and Schulz X-ray method were prepared by the following methods: In order to examine the vicinity just below the surface, the specimen was mechanically ground to about 180 $\mu$ from one surface and chemically polished in a 3 pct HF + $H_2O_2$ solution to about 15 $\mu$ thickness, and finally etched with a 3 pct nital. The interior specimen was prepared by mechanical grinding and chemical polishing of both sides of the intermediate annealed sheets. The etch pit technique, done by Taoka *et al.*,[13] was also used for the thin samples prior to TK examination. Representative areas of the microstructure after the preliminary etch pit analysis were selected and the orientations of primary grains were examined in detail by using TK apparatus.[14] The details of the TK examination and analysis have been reported previously.[15]

## III. EXPERIMENTAL RESULTS

### A. *Comparison of Texture between a Depth of 1/10 the Sheet Thickness under the Steel Surface and Center of the Sheet Thickness of the Intermediate Annealed Sheet*

Figures 1(a) and (b) represent (200) pole figures at a depth of 1/10 the sheet thickness under the steel surface and at the center of the sheet thickness, respectively. As can be seen in Figure 1, the dominant texture components in the vicinity of the steel surface are {410}⟨001⟩, {310}⟨001⟩,

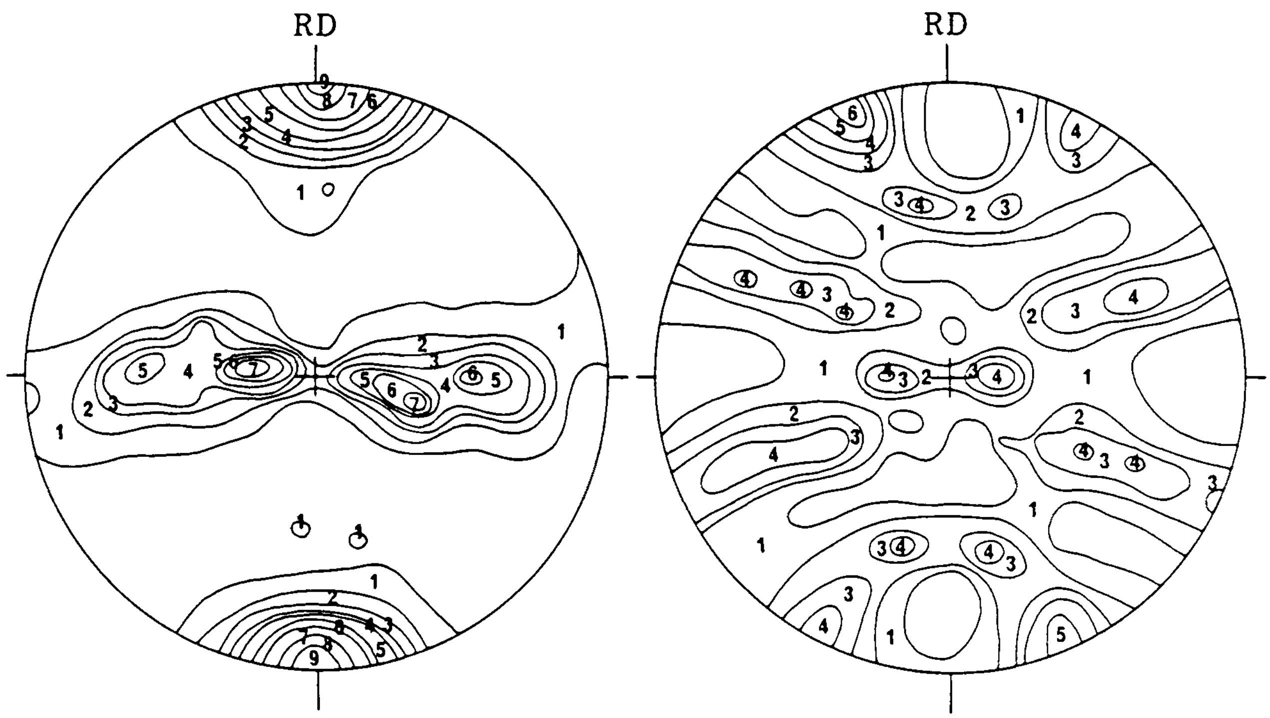



Fig. 1—(200) pole figures at a depth of 1/10 the sheet thickness under the surface and at the center of the sheet thickness of the intermediate annealed sheets containing a small amount of Mo.

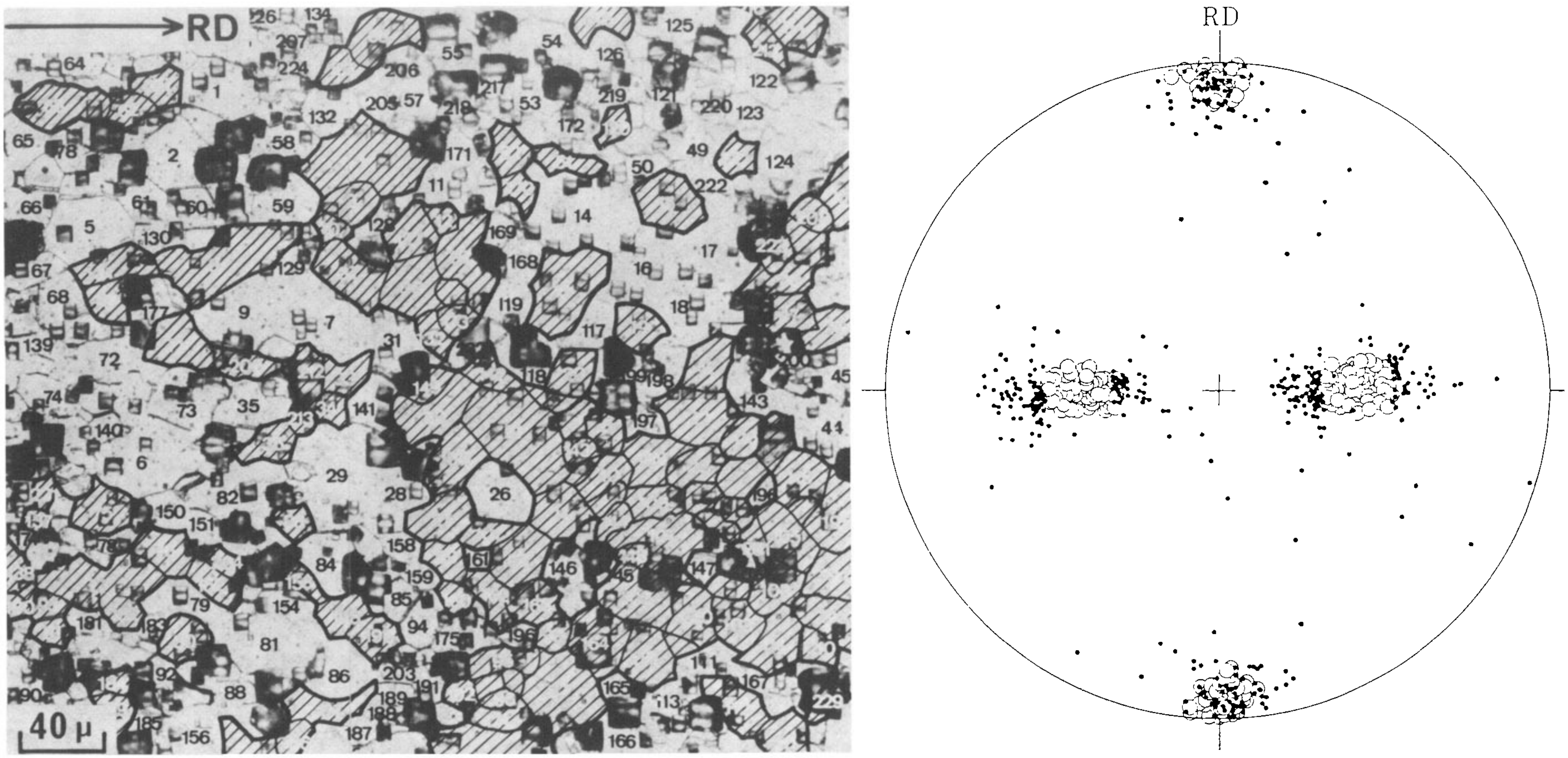



Fig. 2—A montage of the optical micrograph at a depth of 1/10 the sheet thickness under the surface of the intermediate annealed sheet containing a small amount of Mo. This photograph shows the primary grains of {hk0}⟨001⟩ matrix zone, and the right figure shows the stereographic projection of (200) poles obtained from TK patterns of the recrystallized grains in the left photograph.

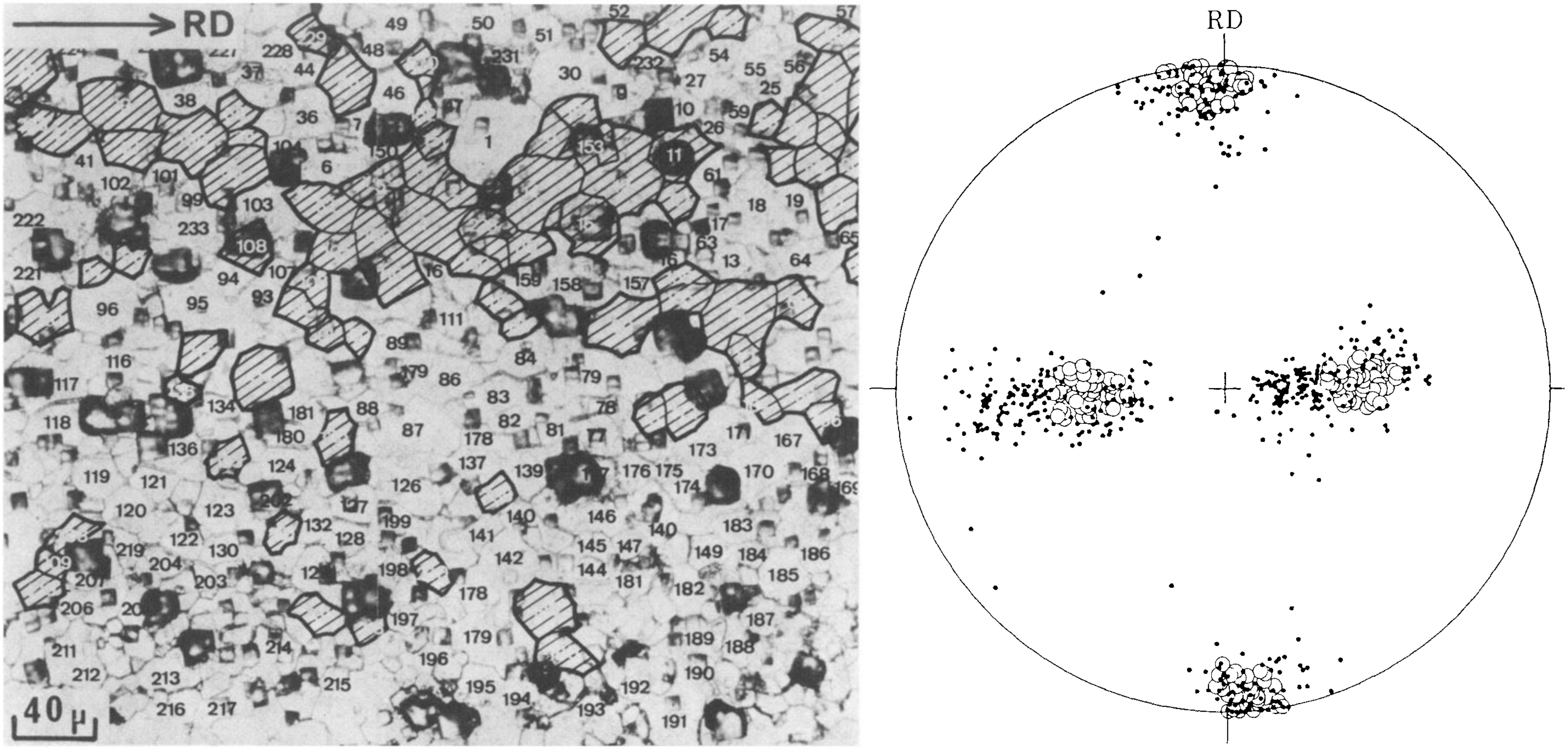
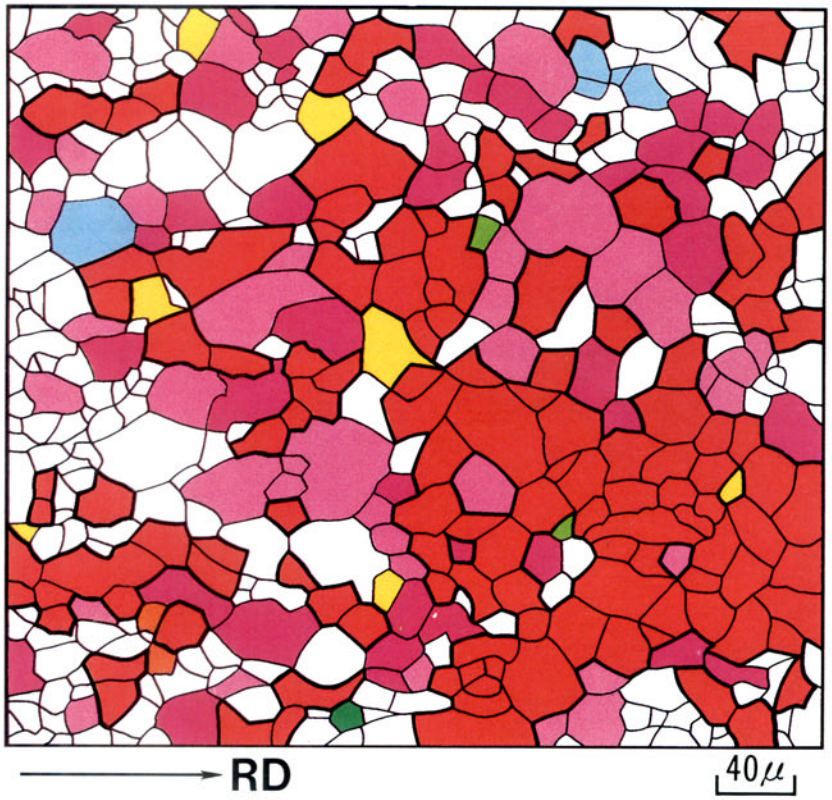



Fig. 4—A montage of the optical micrograph at a depth of 1/10 the sheet thickness under the surface of the intermediate annealed sheet containing a small amount of Mo. This photograph shows the primary grains with strong {hk0}⟨001⟩ and {100}⟨001⟩ matrix zones, and the right figure shows the stereographic projection of (200) poles obtained from TK patterns of the recrystallized grains in the left photograph.




| | Deviation | Av.dia.($\mu$) |
|---|---|---|
| | $(110)\leq 10°$, $[001]\leq 10°$ | 25.3 |
| | $(110)\leq 15°$, $[001]\leq 10°$ | 24.7 |
| | $(110)\leq 10°$, $[001]\leq 15°$ | 16.3 |
| | $(110)\leq 15°$, $[001]\leq 15°$ | 20.5 |
| | $\{210\}\leq 15°$, $\langle 001\rangle\leq 10°$ | 28.7 |
| | $\{200\}\leq 20°$, $\langle 001\rangle\leq 20°$ | 21.9 |
| | $\{200\}\leq 20°$, $\langle 011\rangle\leq 20°$ | —— |
| | $\{111\}\leq 20°$, $\langle 011\rangle\leq 20°$ | 8.2 |
| | $\{111\}\leq 20°$, $\langle 112\rangle\leq 15°$ | 23.1 |

Fig. 3—The schematic illustration of the orientation analysis of the grains of Fig. 2.

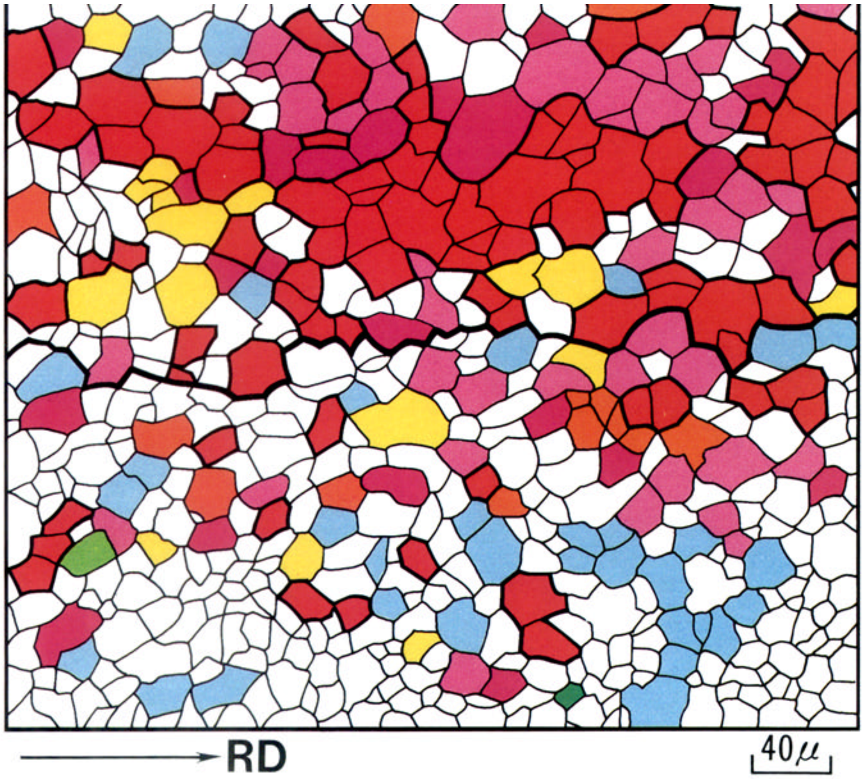
{210}⟨001⟩, and {110}⟨001⟩, and those of the center of the sheet thickness are {200}~{621}⟨012⟩ and {554}⟨225⟩. It is evident that the preferential formation of the {hk0}⟨001⟩ texture containing (110)[001] grains, from which the secondary grains originate, takes place in the vicinity of the steel surface rather than the center of the sheet thickness. Also, in intermediate annealed sheets containing a small amount of Mo[16] the {hk0}⟨001⟩ texture near the steel surface is strengthened and {111}⟨112⟩ and {111}⟨110⟩ textures are weakened due to the addition of Mo.

### B. *TK Examination at a Depth of 1/10 the Sheet Thickness under the Steel Surface of the Intermediate Annealed Sheet*

Figure 2 shows a montage of the optical micrograph of etch-pit and TK analysis taken at a depth of 1/10 the sheet thickness under the steel surface, where {hk0}⟨001⟩ texture is strong. The orientation of individual grains labeled with numbers in Figure 2 was examined in detail by TK technique and plotted in the stereographic projection of (200) poles on the right-hand side of Figure 2. The shaded grains, which are labeled with the white numbers in Figure 2 and plotted with a large circular marking in (200) poles, have the crystallographic orientations within 10 deg from (110)[001] orientation. In order to see a clear tendency from the orientation analysis in Figure 2, a sketch of grain orientations classified by nine colors is illustrated in Figure 3. The average grain diameter is also shown in the table of Figure 3.

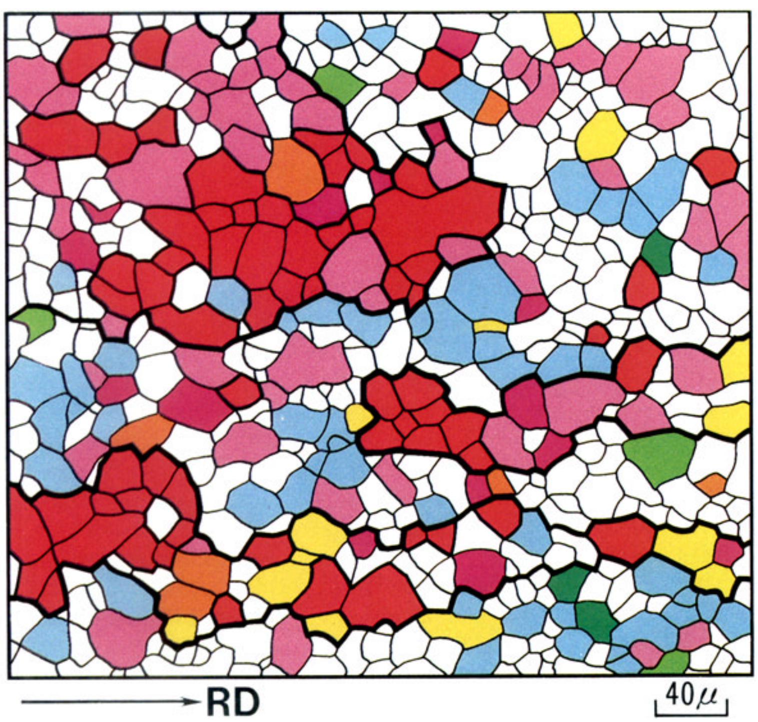
As can be seen in Figures 2 and 3, there are recrystallized (110)[001] grains formed with the colonies of many grains with (110)[001] orientation. In particular, it should be noted that in the sketch of grain orientations in Figure 3 the red-colored grains occupy an extremely large area. The areal fraction of the colonized (110)[001] grains is about 40 pct, although the size of each single (110)[001] grain is comparable to or slightly larger than that of other matrix grains. The fraction of the colonized (110)[001] grains of an intermediate annealed sheet containing a small amount of Mo is approximately 1 to 5 times as large as that with no Mo.[16] This difference in the formation of (110)[001] grains agrees well with the difference between that of hot-rolled Mo added and Mo free steels. Moreover, it should be noted that the formation of {111}⟨112⟩ and {111}⟨110⟩ grains is noticeably decreased due to the addition of Mo.

Figures 4 and 5 show the montages of the optical micrographs of etch-pit and TK analysis taken at a depth of 1/10 the sheet thickness under the steel surface. Also, the orientations of grains in Figures 4 and 5 are plotted in the stereographic projections of (200) poles on right-hand sides of Figures 4 and 5, respectively. Orientation analyses of the grains shown in Figures 4 and 5 are illustrated in Figures 6 and 7, respectively. In Figure 6 there are two matrix zones elongated in the rolling direction: in the top matrix zones the colonies of many (110)[001] grains (colored red) are observed, and in the bottom zone many grains with other orientations (nine other colors) are observed. On the other hand, in Figure 7 there are many matrix zones elongated in the rolling direction with comparatively small widths of 20 to 200 $\mu$, where the colonies of many (110)[001] grains are observed in the {hk0}⟨001⟩ matrix zones, and a few small (210)[001] grains and colonies of several {100}⟨001⟩ grains (colored blue) are observed in the {100}⟨001⟩ matrix zones.




| | Deviation | Av.dia.(μ) |
|---|---|---|
| | (110)≤10°, [001]≤10° | 24.1 |
| | (110)≤15°, [001]≤10° | 22.9 |
| | (110)≤10°, [001]≤15° | 20.3 |
| | (110)≤15°, [001]≤15° | 23.9 |
| | {210}≤15°, <001>≤10° | 21.6 |
| | {200}≤20°, <001>≤20° | 21.8 |
| | {200}≤20°, <011>≤20° | —— |
| | {111}≤20°, <011>≤20° | 15.2 |
| | {111}≤20°, <112>≤15° | 10.7 |

Fig. 6 — The schematic illustration of the orientation analysis of the grains of Fig. 4.




| | Deviation | Av.dia.(μ) |
|---|---|---|
| | (110)≤10°, [001]≤10° | 24.6 |
| | (110)≤15°, [001]≤10° | 21.4 |
| | (110)≤10°, [001]≤15° | 25.8 |
| | (110)≤15°, [001]≤15° | 25.5 |
| | {210}≤15°, <001>≤10° | 24.6 |
| | {200}≤20°, <001>≤20° | 21.4 |
| | {200}≤20°, <011>≤20° | —— |
| | {111}≤20°, <011>≤20° | 23.3 |
| | {111}≤20°, <112>≤15° | 19.9 |

Fig. 7 — The schematic illustration of the orientation analysis of the grains of Fig. 5.

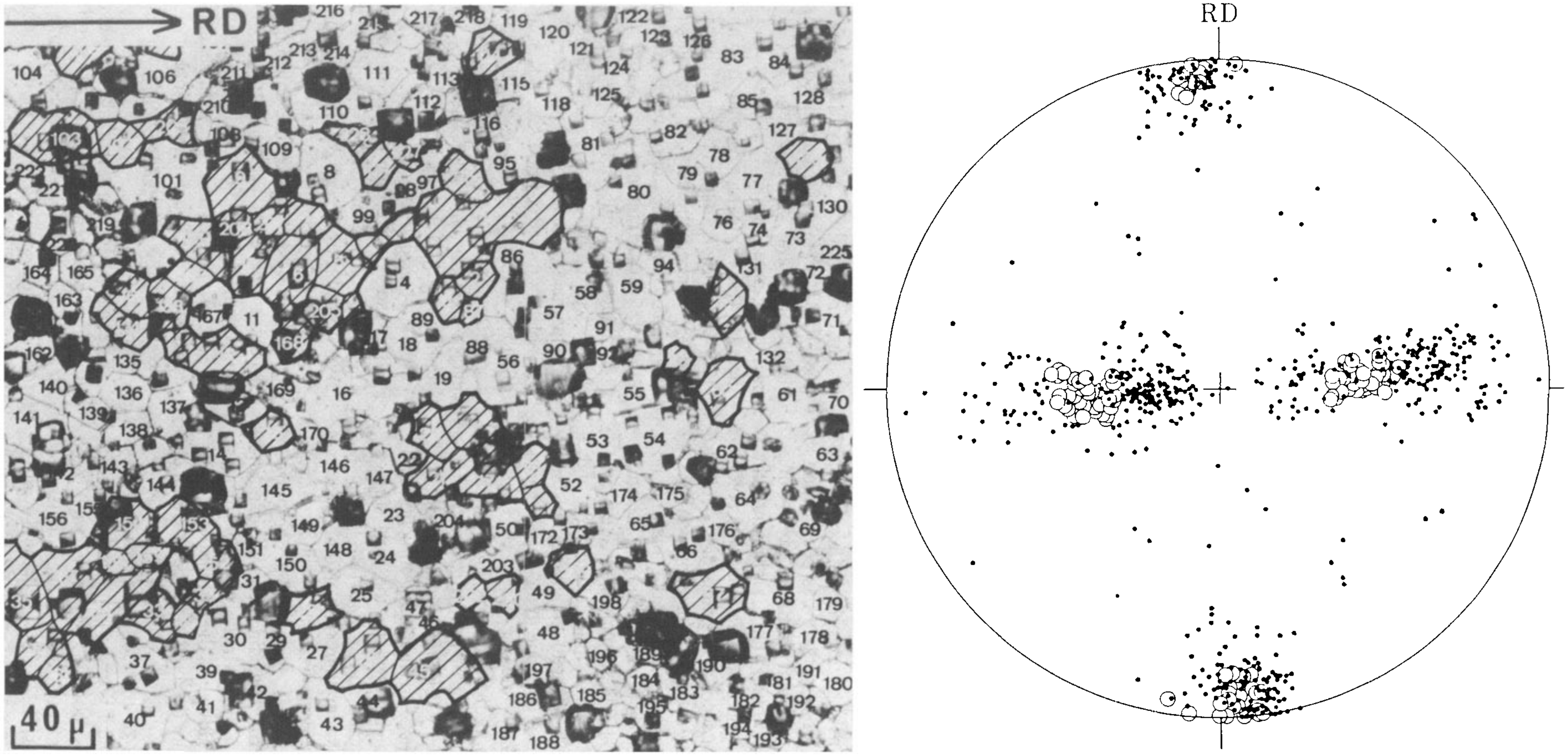



Fig. 5—A montage of the optical micrograph at a depth of 1/10 the sheet thickness under the surface of the intermediate annealed sheet containing a small amount of Mo. This photograph shows the primary grains with small {hk0}⟨001⟩ and {100}⟨001⟩ matrix zones, and the right figure shows the stereographic projection of (200) poles obtained from TK patterns of the recrystallized grains in the left photograph.

From Figures 6 and 7 it should be noted that the formation of (110) [001] grains differs remarkably from the matrix zones and they take place preferentially in the {hk0}⟨001⟩ matrix zones.

### C. *TK Examination of the Central Area of the Intermediate Annealed Sheet*

Figures 8(a) and (b) show a montage of the optical micrograph of the central area observed after the etch pit treatment. The typical grains labeled with numbers in the photograph were examined by TK technique. Figures 9(a) and (b) give the related stereographic projection of (200) poles. Two matrix zones can be seen in Figures 8 and 9: in the top matrix zone there are many recrystallized grains of near {200}⟨013⟩ orientations, and in the bottom matrix zone there are many recrystallized grains having both clockwise and anticlockwise rotations up to 10 deg about the rolling plane normal to the {554}⟨225⟩ orientation (full and dotted lines are marked in Figure 9(b)). There is also the formation of a few small {200}⟨002⟩ grains. However, (110) [001] grains are not found in Photo 4.

## IV. DISCUSSION

### A. *Preferential Formation of (110) [001] Grains near the Steel Surface after the Intermediate Annealing Treatment*

In recent TK examinations[7,8] of hot-rolled silicon steels containing a small amount of Mo, there has been experimental evidence to allow confident assertions concerning the mechanism for the generation of (110) [001] texture; the frequency of generation and the areal fraction of the potential nuclei of the (110) [001] secondary grains in hot-rolled steel containing a small amount of Mo are approximately three times as large as that of hot-rolled steel containing no Mo. Therefore, it is believed that, taking into consideration the inheritance of the (110) [001] secondary nuclei by the structure memory from the original hot-rolled texture, high induction grain oriented silicon steel containing a small amount of Mo has a lower iron loss due to the development of small (110) [001] secondary grains.

In the present investigation of the intermediate annealed sheets containing a small amount of Mo, it is pointed out that there are colonies of many (110) [001] grains inside the defined matrix zones elongated in the rolling direction at a depth of 1/10 the sheet thickness under the steel surface and, although the size of each single (110) [001] grain is comparable to or slightly larger than that of other matrix grains, the colonized (110) [001] grains occupy about 40 pct at the maximum area. The areal fraction of the colonized (110) [001] grains of an intermediate annealed sheet containing a small amount of Mo is approximately 1 to 5 times as large as that with no Mo.[16] However, the formation of colonized (110) [001] grains is not detected in the central area of the sheet.

A schematic illustration of the elongated matrix zones observed in Figures 3, 6, and 7 is given in Figure 10, where the shaded areas exhibit the colonies of the recrystallized grains with (110) [001] orientation. It should be emphasized that the formation of (110) [001] grains is strongly influenced by the elongated matrix zones; in {hk0}⟨001⟩ matrix zones there are many (110) [001] grains and in {200}⟨001⟩ matrix zones there are a few small (110) [001] grains. Also, note that, within the matrix zones containing the colonies of many (110) [001] grains, the (110) [001] grains are surrounded by many {210}~{110} ⟨001⟩ grains and, within the comparatively narrow matrix zones containing the isolated (110) [001] grains, they are surrounded by many {310}~{210} ⟨001⟩ grains. Thus two kinds of matrix zones with (110) [001] grains, which can be inherited by the structure memory from the original hot-rolled texture, have already been found by the detailed TK examination[7,8] of the hot-rolled steel containing a small amount of Mo.

### B. *The Effects of Mo Addition on (110) [001] Grain Formation*

Table I compares the texture formation, the average matrix grain sizes, and the areal fraction of (110) [001] grains of steel containing a small amount of Mo and Mo free. As can be seen in the texture formation of Table I, the intermediate annealed steel containing a small amount of Mo forms strong {hk0}⟨001⟩ texture in the vicinity of the steel surface and {554}⟨225⟩ texture in the central area of the sheet, whereas that of Mo free forms weaker {hk0}⟨001⟩ texture in the vicinity of the steel surface and the texture having {113}⟨332⟩, {522}⟨011⟩, and {200}⟨012⟩ components in the central area of the sheet. It should be emphasized that the texture formation due to the addition of Mo is very profitable for developing the celebrated (110) [001] secondary grains. In the vicinity of the steel surface strong {hk0}⟨001⟩ texture formation containing the colonized (110) [001] grains, from which the (110) [001] secondary nuclei originate, can be seen and in the central area of the sheet {554}⟨225⟩ texture formation, which is related to the {110}⟨001⟩ orientation with the highest rate of growth by rotation of approximately 30 deg about a common ⟨110⟩ axis, can be seen. The importance for the preferential formation of {554}⟨225⟩ texture in a one-stage cold rolling of the high induction grain oriented silicon steel containing AlN and MnS as inhibitors has been described by Matsuo *et al.*[17]

In the next comparison of the average matrix grain sizes of Table I the matrix grain size containing a small amount of Mo is smaller by about 10 pct than that of Mo free. It is considered that the normal grain growth is slightly inhibited by the addition of Mo. The strong inhibition of normal grain growth is very effective in developing the celebrated (110) [001] secondary grains.

Because the preferential formation of the celebrated (110) [001] secondary nuclei prior to the secondary recrystallization treatment is inherited from that of the colonized (110) [001] grains formed in the vicinity of the steel surface during an intermediate annealing, it can be emphasized that the areal fraction of the colonized (110) [001] grains is increased markedly by the addition of Mo. Therefore, it is believed that the high induction grain oriented silicon steel containing a small amount of Mo has lower iron loss due to the development of small (110) [001] secondary grains.

The reasons for hot-rolled silicon steel containing a small amount of Mo forming a strong {110}⟨001⟩ texture near the steel surface are as follows: molybdenum, which is a ferrite forming element in $\alpha$-iron, inhibits local transformations such as $\alpha \rightarrow \gamma + \alpha \rightarrow \alpha$ during hot-rolling. As well, the

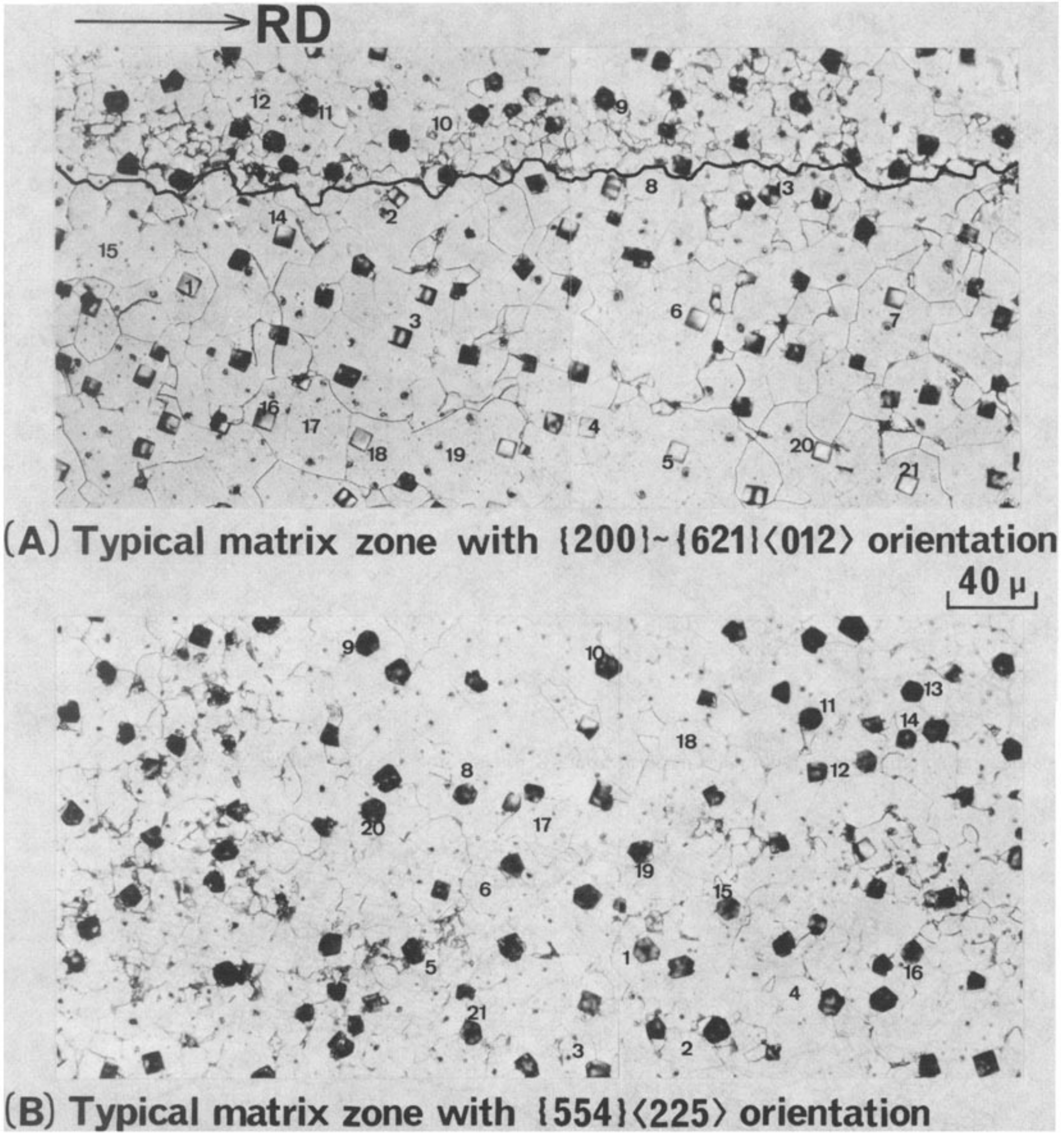



Fig. 8—A montage of the optical micrograph at the central area of the intermediate annealed sheet containing a small amount of Mo.

development of recrystallized grains is much more noticeably retarded and the polygonized {110}⟨001⟩ grains elongated in the rolling direction are more preferentially formed.

Finally, the question remains, how does Mo influence the deformation and recrystallization processes? With respect to these problems a clear evidence has not been obtained yet. However, it is considered that the addition of Mo will be profitable for creating many deformation bands in the {111}⟨112⟩ deformed matrix during first cold reduction and will form preferentially {110}⟨001⟩ primary grains during an intermediate annealing.

## V. CONCLUSIONS

The results obtained are summarized as follows:

1. The average matrix grain size of an intermediate annealed steel containing a small amount of Mo is slightly smaller by about 10 pct than that of Mo free.
2. In the vicinity of the steel surface there are the colonies of many (110) [001] grains in the elongated and well-defined {hk0}⟨001⟩ matrix zones. The areal fraction of the colonized (110) [001] grains is about 40 pct at the maximum value. However, the formation of the colonized (110) [001] grains is not detected in the central area of the sheet.
3. The size of each single (110) [001] grain formed in the vicinity of the steel surface is comparable to or slightly larger than that of other matrix grains.
4. The areal fraction of the colonized (110) [001] grains of an intermediate annealed sheet containing a small amount of Mo is approximately 1 to 5 times as large as that of Mo free.
5. It is believed that the preferential formation of the colonized (110) [001] grains during an intermediate annealing is inherited by the structure memory from the original hot-rolled texture and is noticeably strengthened due to the addition of Mo. Therefore, the high induction grain

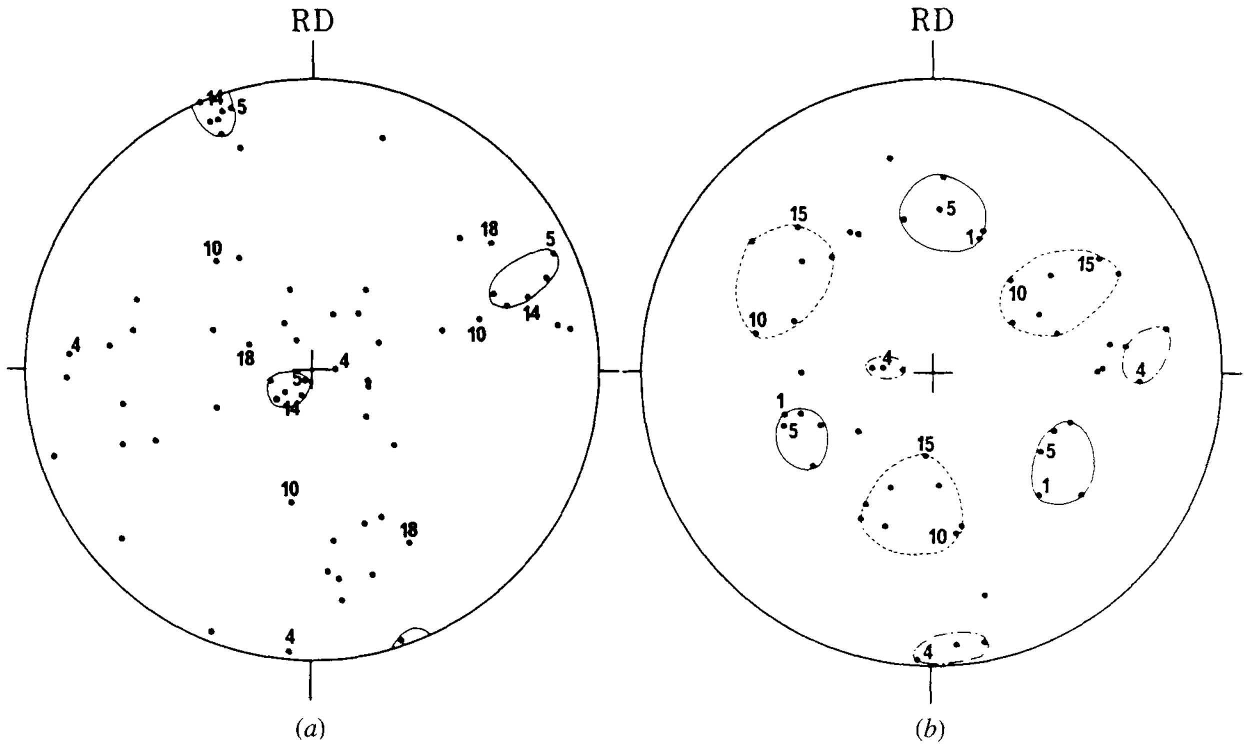



Fig. 9—The stereographic projection of (200) poles obtained from TK patterns from the recrystallized grains in Fig. 8.

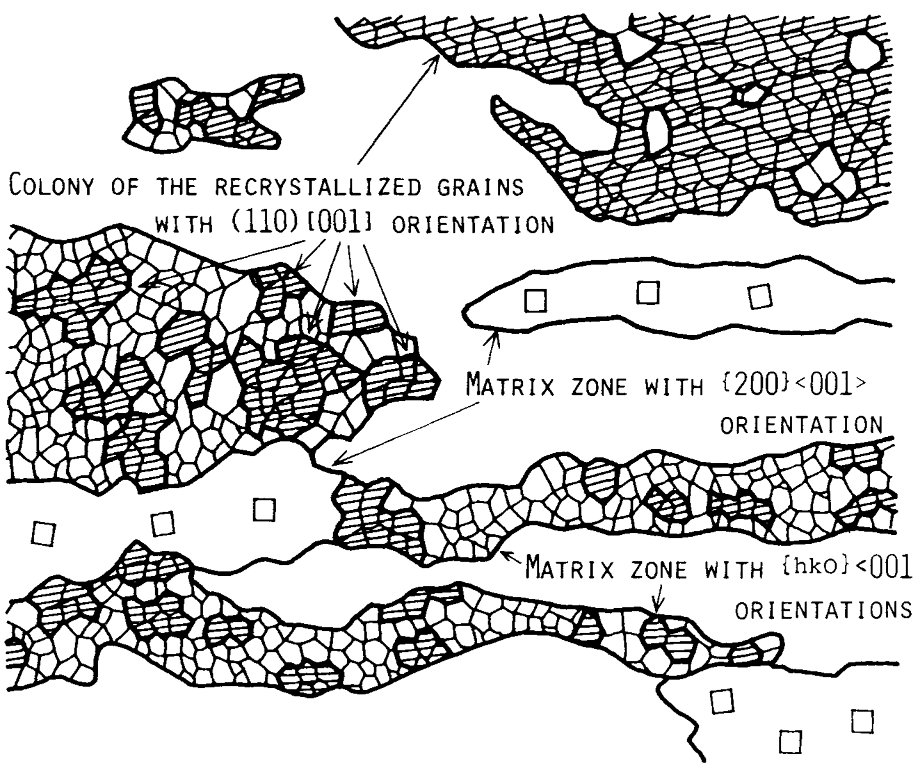



Fig. 10—A schematic illustration of the primary recrystallized grains at a depth of 1/10 the sheet thickness under the surface of the intermediate annealed sheets containing a small amount of Mo.

**Table I. The Comparison of the Texture Formation, the Average Matrix Grain Sizes, and Areal Fraction of (110) [001] Grains of Mo Added and Mo Free Steel.**

| Texture of Matrix Zone | | Average Grain Diameter | Area Occupied by (110) [001] Grains |
|---|---|---|---|
| Surface | Interior | | |
| (A) Mo Added | | | |
| Strong {hko}⟨001⟩ | {200} ~ ⟨621⟩⟨012⟩, {554}⟨225⟩ | 19 μ | 20.7 ~40.2 pct |
| (B) Mo Free | | | |
| {hko}⟨001⟩ | {113}⟨332⟩, {522}⟨011⟩, {200}⟨012⟩ | 21 μ | 8.0 ~22.0 pct |

oriented silicon steel containing a small amount of Mo has lower iron losses due to the development of small (110) [001] secondary grains.

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