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著者	KATAURA Yasuzi
journal or publication title	Science reports of the Research Institutes, Tohoku University. Ser. A, Physics, chemistry and metallurgy
volume	21
page range	159-173
year	1969
URL	http://hdl.handle.net/10097/27494

Grain Structure and Precipitation of Aluminium Nitride in Aluminium and Calcium Deoxidized Low Carbon Steels*

Yasuzi KATAURA

The Research Institute for Iron, Steel and Other Metals

(Received December 3, 1969)

Synopsis

The effects of cold reduction, annealing temperature, heating rate and thermal history before cold reduction on the recrystallization in silicon-deoxidized, aluminium-deoxidized and aluminium-calcium-deoxidized low carbon steels were investigated by means of microscopical observation, hardness test, chemical analysis and low frequency internal friction measurement. The recrystallization behaviour of aluminium-deoxidized and aluminium-calcium-deoxidized steels differed from that of silicon-deoxidized steel, and in the former two steels annealed the structure of elongated grain was obtained as compared with that of equi-axed one in the silicon-deoxidized steel. By means of low frequency internal friction measurement, it was shown that in the steel of elongated grain structure the lowering of dissolved nitrogen occurred during the period from the recovery to an early stage of recrystallization. This would suggest that the nitrogen, at this stage, existed either as preprecipitation clusters with aluminium or as very fine precipitates of AlN. The mechanism of the formation of elongated grain was considered in terms of the effectiveness of AlN precipitation during recrystallization.

I. Introduction

Low carbon sheet steel is the very important material in the steel industry. Small but closely controlled additions of Al and Ti are often made to obtain a certain deep drawing and non-aging property. It has been known(1)~(10) that the inhibition of recrystallization and the development of elongated pancake grain structure in a cold-rolled, aluminium-killed steel can be associated with the precipitation of AlN particles during a recrystallization anneal. Hitherto it has been studied(11) that when molten steel is deoxidized together with aluminium

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* The **1431st** report of the Research Institute for Iron, Steel and Other Metals.

(1) R.L. Rickett, S.H. Kalin and J.T. Mackenzie, Trans. AIME, **185** (1949), 242.
(2) R.L. Solter and C.W. Beattie, ibid., **191** (1951), 721.
(3) W.C. Leslie, R.L. Rickett, C.L. Dotson and C.S. Walton, Trans. ASM, **46** (1954), 1470.
(4) H. Borchers, ZQ. Kim and H.H. Hoff, Arch. Eisenhütt., **35** (1964), 57; ibid., **36** (1965), 311.
(5) R.H. Goodenow, Trans. ASM, **59** (1966), 804.
(6) W. Jolley, J. Iron Steel Inst., **205** (1967), 321.
(7) I.L. Dillamore, C.J.E. Smith and T.W. Watson, Met. Sci. J., **1** (1967), 49.
(8) P.N. Richards, J. Australian Inst. Metals, **12** (1967), 2.
(9) J.T. Michalak and R.D. Schoone, Trans. Met. Soc. AIME, **242** (1968), 1149.
(10) F.A. Hultgren, Blast Furn. Steel Pl., **56** (1968), 149.
(11) T. Ototani, Y. Morooka and Y. Kataura, Sci. Rep. RITU, **A 17** (1965), 326.

and calcium, not only the amount of aluminium oxide in steel is decreased remarkably due to calcium addition, but also retained aluminium is more available for grain refinement and non-aging of steel. The present study was undertaken to see the relationship between the formation of elongated pancake grain structure and the precipitation of AlN in a low carbon, aluminium and calcium-deoxidized steel during recrystallization, and to make more clearly the role of calcium in the calcium-treated steel. To see the behaviour of AlN in the steel, with the exception of chemical analysis, the low frequency internal friction measurement was employed.

II. Specimens and experimental method

3.5 kg of electrolytic iron was melted in an alumina-lined graphite crucible by a high frequency induction furnace. Deoxidization was performed at 1650°C with 0.5% of electrolytic manganese and 0.3% of metallic silicon, and the melt was divided into three parts at tapping, to the first of which any deoxidizer was no more added, to the second of which 0.1% of aluminium was added and to the third of which 0.1% of aluminium plus 1% of Fe–Ca–Si alloy (23.8% Ca, 42.6% Si, Bal., Fe) were added. They were cast in iron mold 30 mm in dia. and 170 mm in length. Thus, three kinds of steel, namely, silicon-deoxidized, aluminium-deoxidized and aluminium-calcium-deoxidized steels were prepared. Chemical compositions of the as-cast specimens are shown in Table 1. These three kinds of ingot were forged into 10 mm square, which after the solution treatment at 1200°C were immediately hot-rolled to 6 mm in thickness through two roll passes with a finishing temperature of about 900°C. Specimens 20 mm by 10 mm were cut from the cold-rolled sheets after 50, 67 and 83% cold reductions. The examination was made of the effects of thermal history prior to cold work, heating rate to the annealing temperature, recrystallization temperature ranging from 500 to 650°C, Vickers microhardness and the precipitation of AlN. For the measurement of internal friction 8 to 10 wire specimens 1 mm in dia. and 200 mm in length were solution-treated in a sealed-quartz tube for 20 min at 1200°C (steel S) and for 3 hr at 1200°C (steel A and C), and then quenched directly from the furnace into the ice water. Immediately after the solution treatment they were reduced 40% into 0.77 mm dia. by cold drawing and then subjected to isothermal annealing at 600

Table 1. Chemical composition of specimens (%)

Steel	Preliminary deoxidants (%)	Final deoxidants (%)	C	Si	Mn	P	S	Al	N
S	Mn 0.5, Si 0.3	—	0.014	0.053	0.14	0.002	0.008	0.004	0.011
A	Mn 0.5, Si 0.3	Al 0.1	0.010	0.041	0.14	0.002	0.008	0.068	0.011
C	Mn 0.5, Si 0.3	Al 0.1, Fe-Ca-Si 1.0	0.016	0.43	0.17	0.002	0.007	0.057	0.012

to 650°C or to isochronal annealing at 100 to 800°C for 3 hr. Kê type torsional pendulum with the frequency of 1.3 cps and the strain amplitude of 7×10^{-5} was used to determine the dissolved carbon and nitrogen in the specimen quenched in ice water. The heating rate for the measurement was 2°C per min and the temperature was measured with the accuracy of ±1°C by the thermocouples of alumel-chromel which were settled in three parts of inner homogeneous heat-exchanger.

III. Experimental results

1. Isothermal recrystallization

Isothermal recrystallization curves for three kinds of steel annealed at 600 and 650°C after 50% cold reduction, based on the hardness of furnace-cooled specimens, are shown in Fig. 1. In the silicon-deoxidized steel S corresponding to the rimmed steel, the hardness decreased within 10 to 30 min and the recrystallization took place in a much shorter time than in aluminium- or aluminium-calcium-deoxidized steels A and C, in which the hardness did not fall for a longer time and the recrystallization proceeded at a slower rate. The marked difference in the shape of these recrystallization curves was found. As far as the effect of temperature on the rate of isothermal recrystallization, the time required for a comparable amount of recrystallization was less the higher the temperature. Fig. 2 shows the isothermal recrystallization curves for steels S and C annealed at 600°C after various cold reductions. By increased cold reduction the stored energy was released at a relatively fast rate so that the recrystallization was followed at an early stage. After cold reductions of 50 and 67%, the silicon-deoxidized steel

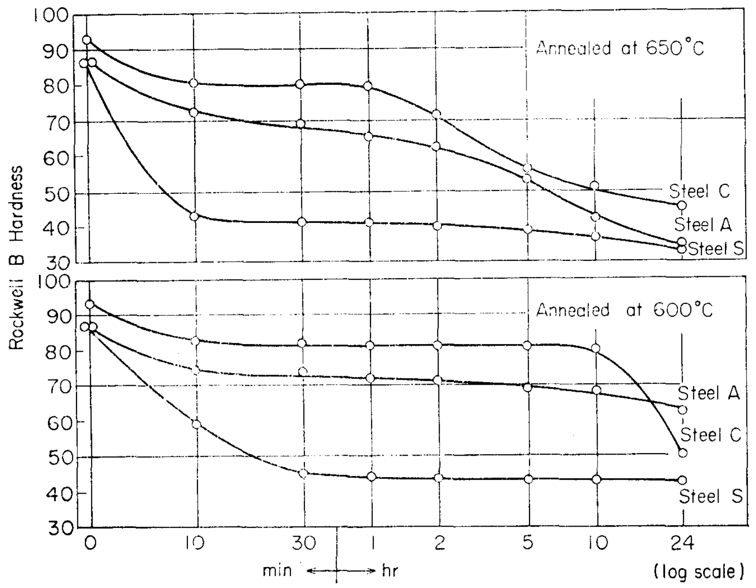

Fig. 1. Rate of isothermal recrystallization of rimmed steel S, aluminium-deoxidized steel A and aluminium-calcium-deoxidized steel C annealed at 600 and 650°C after 50 pct cold reduction.

recrystallized in a much shorter time than the aluminium-calcium-killed steels. In the latter steel C cold-reduced 83%, however, the recrystallization proceeded at a relatively fast rate. The microstructure of fully recrystallized specimens of silicon-deoxidized, aluminium-deoxidized and aluminium-calcium-deoxidized steels was characterized by the grain structure as follows: in the first the grain was equiaxed whereas in the latter two steels it was elongated both in the longitudinal and in the transverse section. The amount of grain elongation in both sections of sheet surface based on grain counts made by the intercept method was 2 to 2.4 in aluminium-deoxidized steel and 2.8 in aluminium-calcium-deoxidized steel. These steels were cold-reduced 50%, annealed at 600°C for 24 hr and grain elongations were measured in the annealed state. In general, the amount of grain elongation developed by a given amount of cold reduction was more in steel C than in steel A. It may be not reasonable to assume from this fact alone that the deep drawability of sheet steel C deoxidized with aluminium and calcium will be more favourable than that of sheet steel A deoxidized only with aluminium. Nevertheless, the improvement of the deep drawability in a lower silicon sheet steel deoxidized with aluminium and calcium will be expected from the report(12) that an addition of calcium improves the cleanliness of the sheets very effectively, varying the amount, the form and the distribution of the nonmetallic inclusions in the sheets, so that the improvement of the cleanliness results in a rise of the impact and toughness properties of the steel. By the recrystallization annealing, aluminium- and aluminium-calcium-deoxidized steel sheets became of elongated grain structure both in the longitudinal and in the transverse section. This

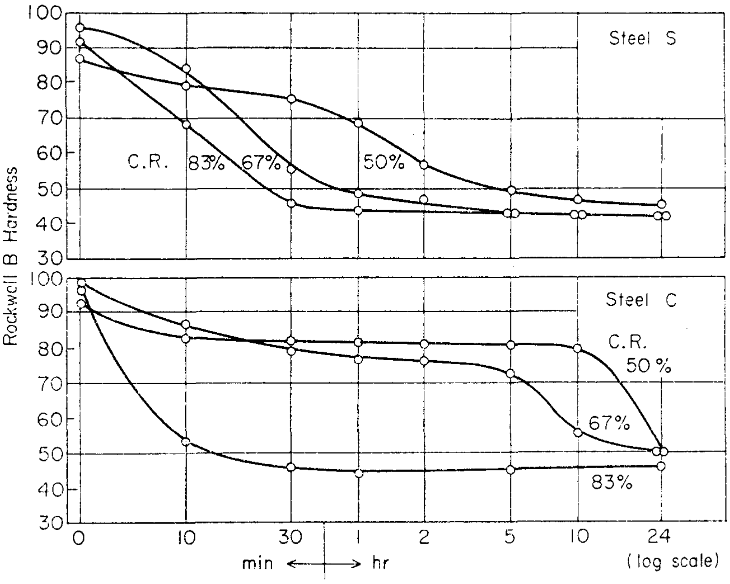

Fig. 2. Rate of isothermal recrystallization of rimmed steel S and aluminium-calcium-deoxidized steel C annealed at 600°C after 50, 67 and 83pct cold reduction.

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(12) M. Wahlster et al., Radex-Rdsch. (1969), H. 2, 478.

appears to be associated with a slower rate of recrystallization, which will be due to the retardation of the annihilation of dislocations introduced by cold work and also to the retardation of the nucleation rate of recrystallization. If a metal contains a material mechanically obstructing grain growth and if the grain boundary migration occurs alternatively by recrystallization anealing, it may develop grains elongated in the direction of rolling. The present experiments suggest that the substance mechanically obstructing grain growth seems to be the precipitate of AlN. Since it was presupposed that the recrystallization behaviour had to be remarkably influenced by the existence of the second phase such as AlN, and that the effectiveness of AlN precipitate might be influenced by the rate of heating in annealing and by the thermal history of the sheet steel before cold reduction, the following experiments were carried out.

2. Effect of heating rate of annealing

Steels S and C cold-reduced 50% were used and annealed at two kinds of heating rate. The one was a rapid heating to 650°C, and the other was a slow heating to 650°C at the uniform rate of 50°C per hr above 400°C. Isothermal recrystallization curves for these two kinds of steel at 650°C, based on the hardness of furnace-cooled specimens, are shown in Fig. 3. A remarkable difference in the change of hardness due to the rate of heating during recrystallization of steel S was not almost observed but the slowly heated specimen softened a little more in the fully recrystallized state than the rapidly heated one. On the other hand, steel C showed a noticeable difference in hardness during recrystallization, and it seemed that the recrystallization process was affected by the rate of heating. Primary recrystallization comprises a nucleation and a growth processes, and under certain conditions renewed grain growth occurs on further increasing temperature. When the specimen is heated slowly the internal energy stored by cold work may be released relatively fast during the recrystallization process and tends to decrease the grain boundary energy. That the hardness was not reduced for a long time in the specimen heated rapidly would be due to a relatively slow rate of the nucleation of recrystallization caused by the retardation of the

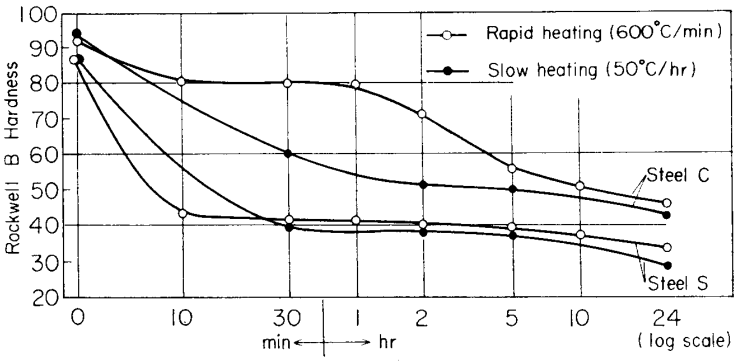
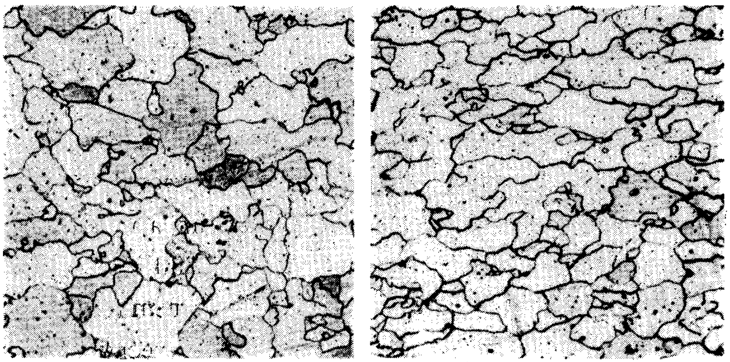


Fig. 3. Effect of heating rate on isothermal recrystallization of rimmed steel S and aluminium-calcium-deoxidized steel C annealed at 650°C after 50 pct cold reduction.

precipitation of AlN; in fact, the hardness curve showed finally a rather sharp drop in the moment a renewed grain was generated. The elongated pancake grain structure after recrystallization of steel C heated slowly is shown in Phot. 1.

3. Effect of thermal history prior to cold reduction

To know the recrystallization behaviour of the steel which was not solution-treated at 1200°C and contained AlN before cold reduction, the influence of the intermediate heat-treatment prior to cold reduction on final grain structure was examined. Four kinds of specimen C-1, C-2, C-3 and C-4 were secured from a hot-rolled material 10 mm square deoxidized together with aluminium and calcium,



Rolling plane Longitudinal section

Phot. 1. Pancake structure of aluminium-calcium-deoxidized steel annealed 24 hr at 650°C when heated slowly at 50°C per hr from 400°C after 50pct cold reduction. 5% Nital etch. × 63

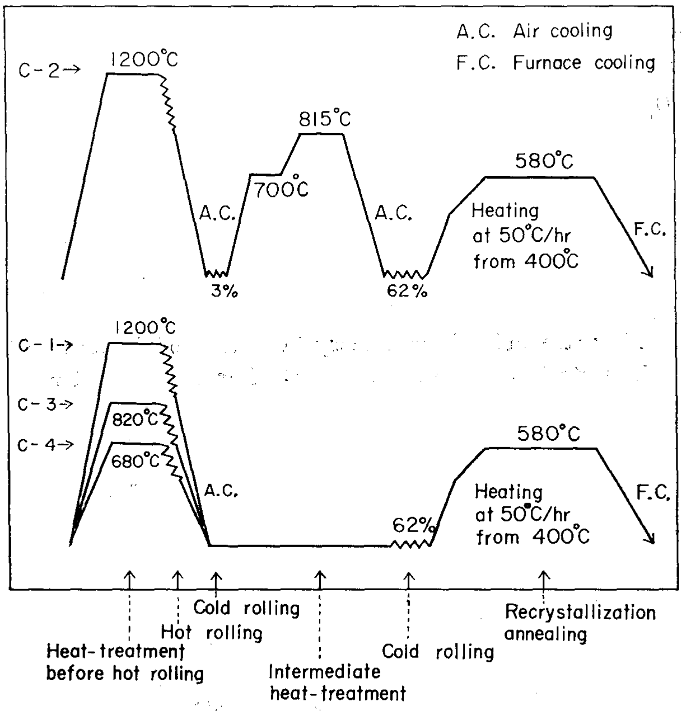


Fig. 4. Schematic cycles of various thermal treatment used for aluminium-calcium-deoxidized steel C-1 to C-4.

and the recrystallization annealing was performed according to the thermal history as shown in Fig. 4. Specimen C-2 was solution-treated at 1200°C for 30 min and at this temperature directly hot-rolled to 6 mm in thickness. Further, in order to precipitate AlN before cold reduction, the specimen was slightly cold-reduced 3% and annealed at 700°C for 1 hr plus 815°C for 1 hr. Specimen C-1 was subjected only to the solution treatment at 1200°C before cold reduction. Specimens C-3 and C-4 were heat-treated respectively at 820 and 680°C for 30 min without solution treatment at 1200°C, and hot-rolled to 6 mm in thickness.

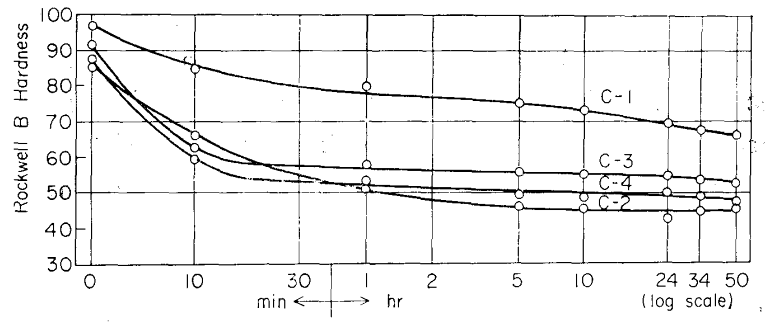
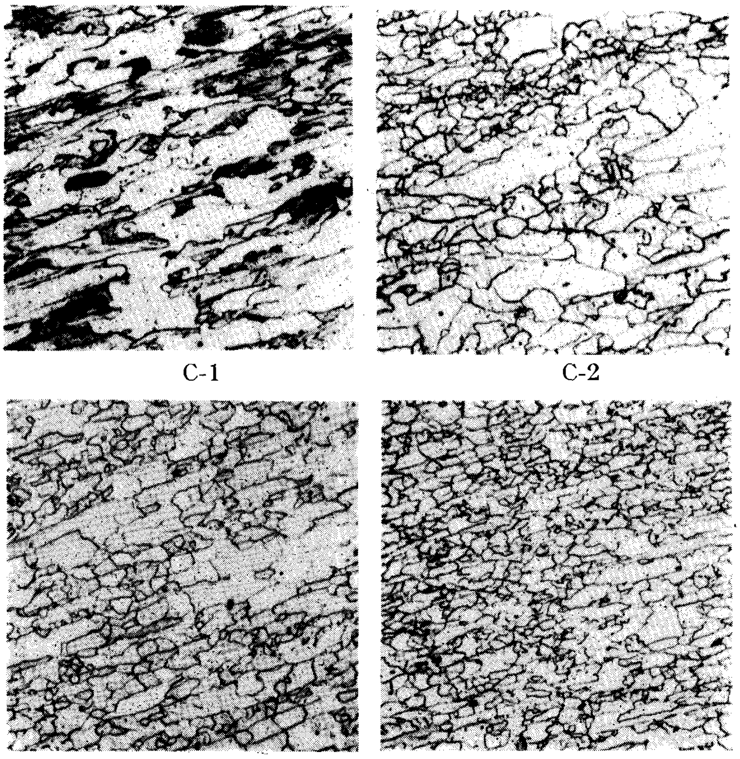


Fig. 5. Effect of thermal treatment before cold rolling on isothermal recrystallization of aluminium-calcium-deoxidized steel C-1 to C-4 annealed at 580°C after 62pct cold reduction.


C-1 C-2

C-3 C-4

Longitudinal section

Phot. 2. Effect of prior heating on grain structure of aluminium-calcium-deoxidized steels annealed 50 hr at 580°C after 62 pct cold reduction. 5% Nital etch. × 63

Following intermediate heat-treatments these four specimens were cold-reduced 62% and isothermally annealed at 580°C after slow heating at the rate of 50°C per hr from 400°C. Fig. 5 shows isothermal recrystallization curves for these four specimens at 580°C, with reference to the hardness of furnace-cooled specimens. Of the specimens which showed Rockwell B hardness nearly 90 in cold-rolld state specimens C-2, C-3 and C-4 showed a sharp drop of hardness after the anneal for 1 hr and the recrystallization in them proceeded at a relatively fast rate. On the other hand, specimen C-1 showed a rather gradual drop of hardness, and the recrystallization proceeded partially without lowering Rockwell B hardness more than 67, even though the annealing time was 50 hr. Microstructures of four specimens annealed at 580°C for 50 hr were as shown in Phot. 2. Specimens C-2, C-3 and C-4 recrystallized fully, softened rapidly and were of nearly equiaxed grain structures, particularly of fine grain in specimens C-3 and C-4. In specimen C-1, however, the grains appeared elongated as can be inferred from the retardation of softening and from the microstructure on the way of recrystallization. When specimen C-1 was annealed at 650°C for 24 hr, the grains were recrystallized fully and elongated both in the lengthwise and in the transverse direction of the sheet.

The foregoing experiment shows that the thermal history prior to cold reduction is an important factor in determining whether the grains in the aluminium- or in the aluminium-calcium-deoxidized steel after annealing will become elongated or equiaxed, and that the solution treatment of AlN before cold reduction is a necessary factor to obtain elongated pancake grains. Fig. 6 shows the different amounts of AlN detected chemically by Beeghly method(13) in the specimens ranging from the as-cast to the recrystallized state. Specimen C-2 heat-treated intermediately, specimen C-3 heat-treated at 820°C and specimen C-4 heat-treated at 680°C before hot rolling precipitated 0.027 to 0.030% of AlN

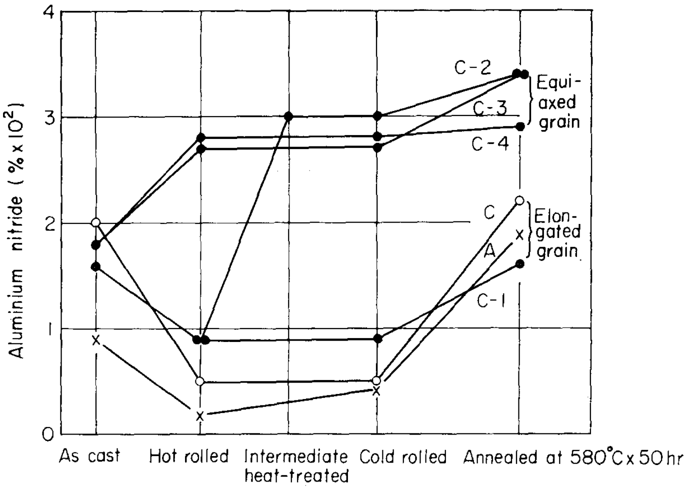


Fig. 6. Effect of thermal history on the aluminium nitride detected by chemical analysis. A: Aluminium-deoxidized steel, C and C-1 to C-4: Aluminium-calcium-deoxidized steel

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(13) H.F. Beeghly, Anal. Chem., **21** (1949), 1513.

in the state of cold rolling. The recrystallized grains in these specimens after annealing were definitely equiaxed, even though the amount of AlN precipitate in the annealed state increased more than in the cold-rolled state. On the other hand, in specimens A, C and C-1 solution-treated at 1200°C the amount of AlN detected after hot rolling decreased remarkably below 0.009%, which could be appreciated as undissolved AlN on the basis of the solubility product by Leslie et al.(3) However, the amount of AlN detected in these specimens after the recrystallization tended to increase from 0.016 to 0.022%, and all specimens were of elongated grain structure. From this experiment it was assumed that a necessary factor to form the elongated pancake grain structure in the steel deoxidized with aluminium and calcium was to perform the solution treatment of AlN in austenite before cold reduction and to control AlN precipitating from supersaturated solid solution during recrystallization. If AlN precipitates in the grain boundary or in the grain interior before cold reduction, it will be expected that the recrystallization proceeds at a relatively fast rate, and that the final recrystallized grains are equiaxed rather than elongated.

4. Internal friction

To know the behaviour of the nitrogen during recrystallization, wire specimens 1 mm in dia. were isothermally annealed in vacuum-sealed quartz glass ampoules at 600 and 650°C, and the amounts of carbon and nitrogen dissolved were measured at various stages during annealing by the internal friction method. The result of the silicon-deoxidized steel S annealed at 600°C after solution treatment at 1200°C for 20 min and 40% cold reduction is shown in Fig. 7. Ferrite grain size of the specimen solution-treated was about 130 μ. The height of the dissolved (C+N) peak $Q_{\max}^{-1}$ after cold reduction was 6×10^{-3} and with increasing annealing time Snoek peak increased gradually to 8.3×10^{-3} after 22 hr anneal corresponding to 100% of recrystallization. A gradual increase of Snoek peak

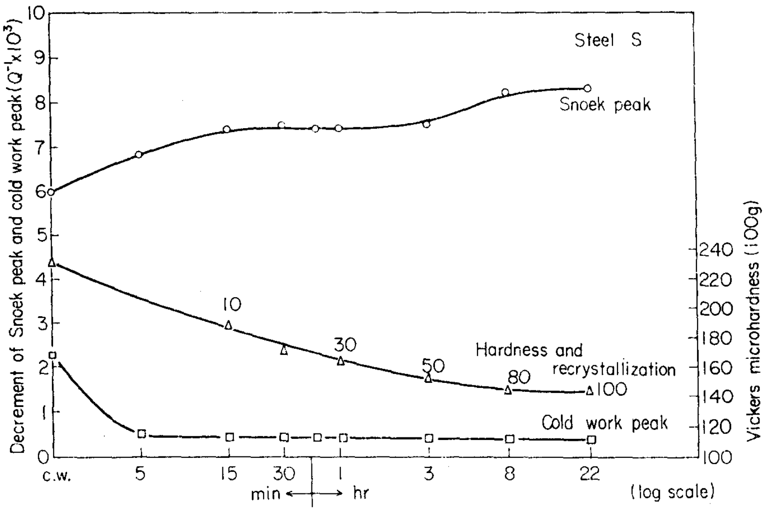

Fig. 7. Snoek peak, cold work peak and hardness of rimmed steel S solution treated at 1200°C and after 40 pct cold reduction depending upon the annealing time at 600°C.

suggests the compensation of the dissolution and the precipitation of interstitial atoms. The Vickers microhardness decreased gradually through recrystallization process. Numerical values noted on the hardness curve showed a volume per cent recrystallized, which was measured by microscopical observation. The cold-work peak observed in the vicinity of 220°C decreased very rapidly in a short time after the isothermal annealing and came down nearer to horizontal level as the annealing time was increased. The lowering of the cold-work peak at an early stage will be attributed to the precipitation of interstitial atoms such as carbon or nitrogen on the dislocations, and also to the rearrangement of dislocations. Fig. 8 is the result of aluminium-deoxidized steel A annealed at 600 or 650°C after solution treatment at 1200°C for 3 hr and 40% cold reduction. What differed from the result on steel S was the fact that with increasing annealing time both Snoek peaks decreased gradually to 2×10^{-3} followed by a rapid increase at 650°C anneal and by a slow increase at 600°C anneal for 19 hr, respectively. The decreasing tendency of Snoek peak which was observed from 0.5 to 8 hr of the annealing time suggests the occurrence of a precipitation during the period from the recovery to the beginning of recrystallization. It was assumed that the decrease of Snoek peak would be due to the lowering of dissolved nitrogen depending upon AlN precipitation. The fact that the rise of Snoek peak was observed, during the recrystallization, rapidly from 30 hr of annealing time, would be due to a dissolution of carbon which was trapped on grain boundaries or formed Cottrell atmosphere around the dislocations, since with the progress of recrystallization the annihilation of dislocations would occur. When the internal friction curves were resolved into two peaks of carbon and nitrogen with the equation(14),

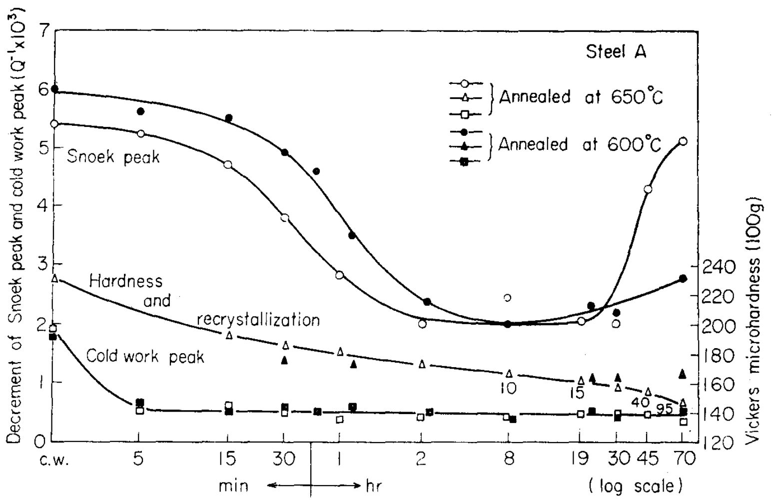

Fig. 8. Snoek peak, cold work peak and hardness of aluminium-deoxidized steel A solution treated at 1200°C and after 40 pct cold reduction depending upon the annealing time at 600 or 650°C.

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(14) W. Wepner, Arch. Eisenhütt., **27** (1956), 449.

the rise of Snoek peak appearing at the last stage was nearly equivalent to the height of the resolved carbon peak. As for the cold-work peak the curves were almost similar in shape to that shown in Fig. 7. Fig. 9 is the result of the aluminium-calcium-deoxidized steel C annealed at 600°C after solution treatment at 1200°C for 3 hr and 40% cold-reduction. Ferrite grain size of the specimen solution-treated was about $130\,\mu$, being the same as those of specimens A and

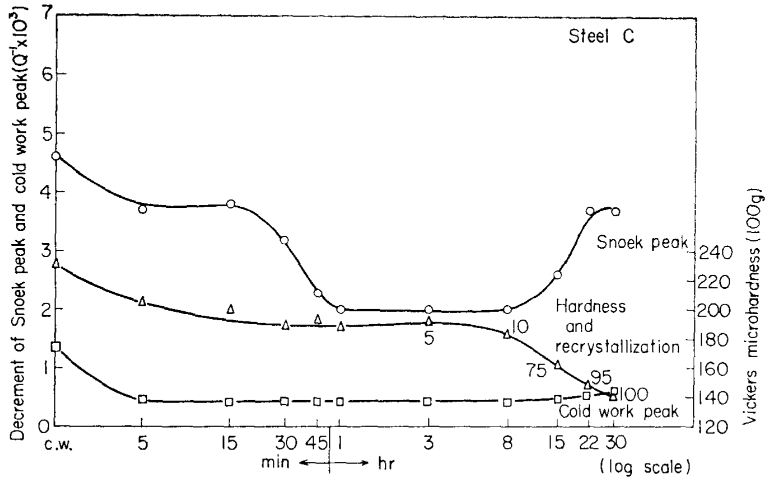

Fig. 9. Snoek peak, cold work peak and hardness of aluminium-calcium-deoxidized steel C solution treated at 1200°C and after 40 pct cold reduction depending upon the annealing time at 600°C.

S. It should be noted that the curves were similar in shape to that shown in Fig. 8, which showed mainly the dependence of dissolved nitrogen. At an early stage of the annealing, the amount of dissolved carbon and nitrogen tended to lower owing to their precipitation on grain boundaries and on dislocations introduced by 40% cold reduction. With increasing annealing time Snoek peak decreased remarkably from the recovery to the beginning of recrystallization to 2×10^{-3}. This would be attributed to the lowering of dissolved nitrogen, based on AlN precipitation occurring preferentially at grain boundaries prior to cold-work or at the sites of the subboundaries. The hardness curve showed an initial gradual drop, then a nearly horizontal portion and finally after annealing for 8 hr a rather sharp drop due to a rapid recrystallization. The rise of Snoek peak observed at the final stage after annealing of 8 hr could be appreciated due to the dissolved carbon as mentioned above, because Snoek peak temperatures observed at this stage were 40 to 43°C.

As can be seen from the result on specimen C-2 shown in Fig. 6, the specimen solution-treated and intermediate-heat-treated above A_1 point precipitated AlN mainly before cold reduction. Here, the effect of the intermediate heat-treatment on the recrystallization behaviour was examined by the internal friction. The heat-treatment after solution treatment at 1200°C for 3 hr was performed at 700°C for 1 hr plus 815°C for 1 hr air cooling, then cold-reduced 40% and annealed at 600°C. The result is shown in Fig. 10. Snoek peak and the cold-work peak in the cold-worked state showed relatively lower levels as compared with that without intermediate heat-treatment as can be seen in Fig. 9. This would be due to the lowering of dissolved nitrogen caused by the precipitation of a greater part of AlN during intermediate heat-treatment. Snoek peak showed an initial gradual drop to 2×10^{-3} and then a reversed gradual rise to 3.5×10^{-3} after 46 hr anneal, the recrystallization proceeding at a relatively fast rate. It was assumed that

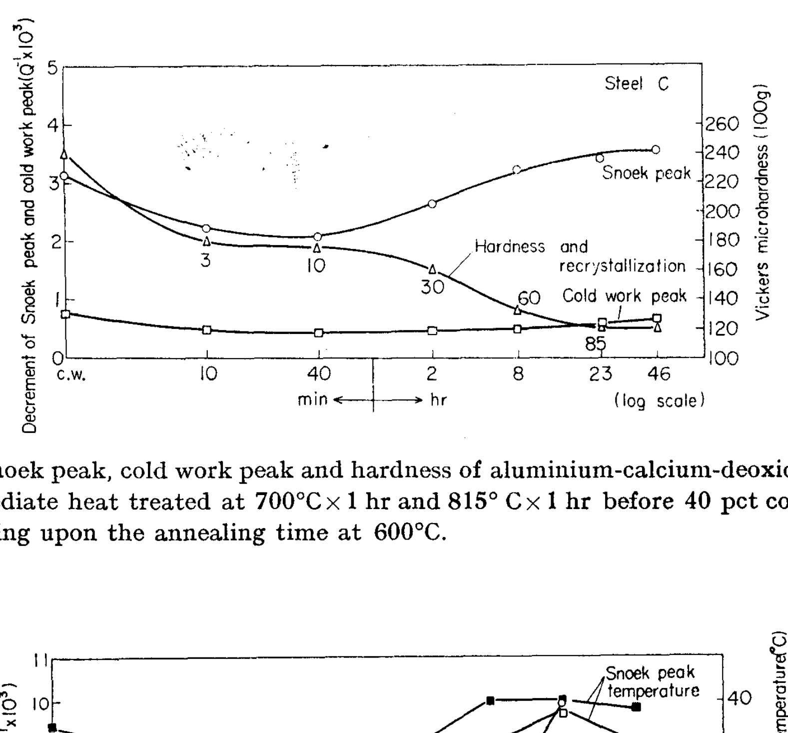

Fig. 10. Snoek peak, cold work peak and hardness of aluminium-calcium-deoxidized steel C intermediate heat treated at 700°C × 1 hr and 815° C × 1 hr before 40 pct cold reduction depending upon the annealing time at 600°C.

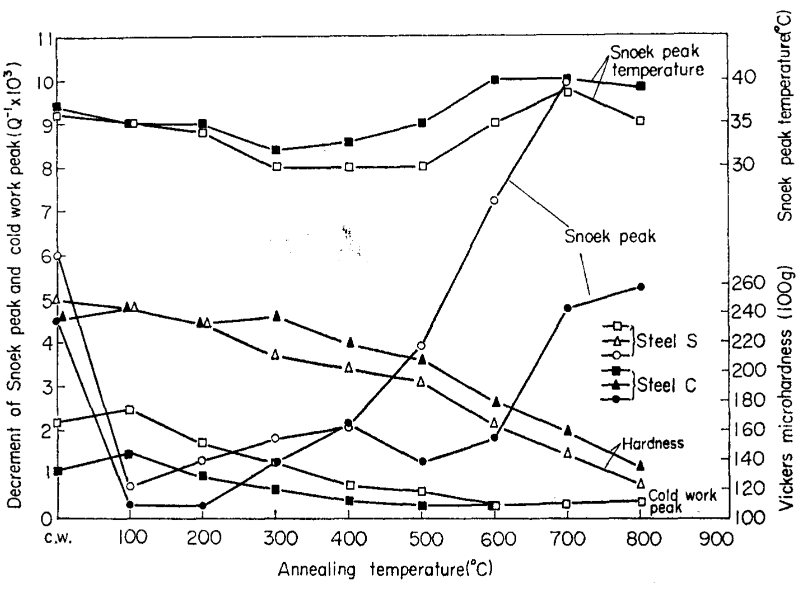

Fig. 11. Snoek peak, Snoek peak temperature, cold work peak and hardness of steel S and steel C solution treated at 1200°C and after 40 pct cold reduction depending upon the annealing temperature for 3 hr.

the gradual increase of Snoek peak observed during recrystallization would be due mainly to the solute carbon originated from the annihilation of dislocations, since Snoek peak temperatures were 40°C corresponding to carbon peak in all specimens annealed above 10 min.

The comparable data of the isochronal annealing at 100 to 800°C for 3 hr of steels S and C are shown in Fig. 11. It should be noted that a remarkable decrease to 3 and 7×10^{-4} of Snoek peak was recognized at 100°C in both steels. This would be caused mainly by locking or precipitating of solute atoms, carbon and nitrogen, on the dislocations. With increasing annealing temperature above 200°C Snoek peak tended to increase due to the increase of carbon and nitrogen solubility in α iron and to the rearrangement of dislocations. This increase was more remarkable in steel S. On the other hand, in steel C Snoek peak was lowered at 500°C temporarily and increased again from 600°C anneal. Different behaviours among these steels seemed to be related to their content of solute carbon and nitrogen. In particular, the lowering of solute atoms in specimen C at 500°C anneal suggests the formation of preprecipitation clustering of AlN in a form which is not detectable chemically by the Beeghly method. Since in the annealing above 600°C Snoek peak temperature in this steel shifted to 40°C of carbon peak, the increase of solute atoms would be attributed mainly to solute carbon. The cold-work peak of both steels decreased from 100°C anneal and this decrease was more remarkable in steel C. The hardness curves of both steels showed an initial gradual drop and then a drop at fast rate above 500°C. Samples of both steels annealed at 700°C were recrystallized fully.

Finally, to compare the solubility of carbon plus nitrogen in α iron, wire specimens of three kinds of steel S, A and C were used for internal friction measurement. They were solution-treated at 1200°C to dissolve the precipitates, after cold reduction of 40% and recrystallization at 650°C annealed in vacuum-sealed quartz glass ampoules at 650, 600 and 500°C for 15 hr and then quenched in ice water. Fig. 12 shows that Snoek peak Q_{max}^{-1} is proportional to carbon plus nitrogen solubility depending upon temperature. Of three steels, steel C deoxidized with aluminium and calcium revealed a least solubility. This effect will be due to the fixing of dissolved nitrogen, which is presumed to form the AlN

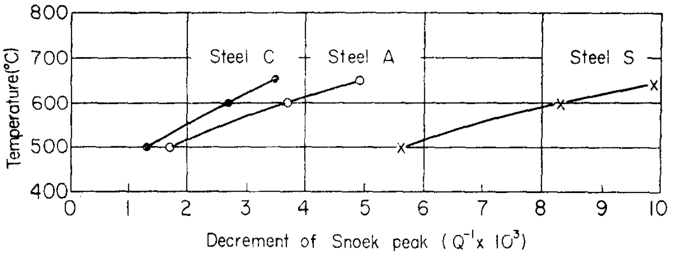

Fig. 12. Relation between the Snoek peak Q_{max}^{-1} and the annealing temperature for 15 hr of steel S, steel A and steel C solution treated at 1200°C, 40 pct cold reduced and after recrystallization at 650°C.

precipitate. From this point of view it is inferred that the aluminium-calcium-deoxidized steel will be possible to possess characteristics of the non-aging steel, considering in connection with the results of previous work examined with these three kinds of steel(15).

IV. Consideration

It has been known from many works on the recrystallization of cold-worked aluminium-killed steels that the retardation of recrystallization, the development of elongated pancake grain structure and the characteristic recrystallization texture can be associated with the precipitation of AlN at the grain boundaries prior to cold work, on the boundaries of the growing recrystallizing grains and on the subboundaries. The effectiveness of AlN to form elongated grains seems to be influenced by the thermal history before cold reduction and the rate of heating in recrystallization annealing. In relation to the precipitation of AlN, it should be noted that the way, the form and the size of precipitation and the distribution of AlN during recrystallization are of very importance in the formation of elongated grains. Recently, it has been ascertained by Borchers et al.(4) that the increasing of AlN precipitate detected by the chemical analysis in the aluminium-killed steel annealed at 600°C after cold reduction of 75% occurs after complete recrystallization, but not at an early stage of recrystallization. This is in good agreement with the present data that the amount of AlN precipitate is low before cold rolling and high after recrystallization to obtain elongated grain structure in the steels deoxidized with aluminium and calcium annealed at 580°C after cold reduction of 62%. If AlN precipitate during recrystallization plays an important role in forming elongated pancake grain structure, they would have a principal influence upon the nucleation and growth of recrystallized grains and may exist in a form which can not be detected by the chemical method. According to Keh et al.(16) an average diameter of separable AlN particles is about 2×10^{-6} cm and aluminium and nitrogen in supersaturated solid solution can be nucleated at subboundaries before the cells have grown to form recrystallized grains, so it is important to know more clearly the preprecipitation cluster of AlN prior to the chemical separation. Such an AlN precipitation would inhibit the nucleation and growth of recrystallized grains having a close concern with the formation of (111) [110] recrystallization texture component,(5),(9),(17) and then growing AlN particles would form a mechanical barrier to the growth of recrystallized grains in the direction vertical to the rolling plane, so that the restraining action of grain boundary migration would easily elongate the grain boundaries which are parallel to the rolling plane.

As can be seen in Fig. 11, the tendency to decrease and to reverse Snoek peak

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(15) W. Köster, W. Horn und Y. Kataura, Arch. Eisenhütt., **37** (1966), 1017.

(16) A.S. Keh, W.C. Leslie and G.R. Speich, *Precipitation on Substructure in Iron-Base Alloys*, (1962), 13.

(17) Z. Kubodera and Y. Inagaki, Bull. Japan Inst. Metals, **7** (1968), 383.

and to decrease the cold-work peak depending upon annealing temperature was nearly in agreement with the results obtained by using commercial steels by Köster et al.(18) and Aoki et al.(19) The decrease of dissolved carbon and nitrogen at 100°C anneal would be due to the diffusion of solute atoms along dislocations. With increasing annealing temperature above 100°C Snoek peak increased gradually according to solubility change of dissolved carbon and nitrogen. In the steel deoxidized with aluminium and calcium unlike the steel deoxidized with silicon, a drop of Snoek peak from 400 to 600°C was recognized. This would presumably correspond to the stage of forming preprecipitation cluster of AlN(5),(9) so fine that it will be impossible to confirm it even by electron microscopy. It was found that the technique of internal friction measurements was a considerably effectual method to detect AlN precipitate at an early stage of recrystallization annealing.

Summary

The effect of cold reduction, heating rate to the annealing temperature and thermal history prior to cold reduction on the recrystallization in low carbon silicon-deoxidized aluminium-deoxidized and aluminium-calcium-deoxidized steels were examined by means of microscopical observation, hardness, chemical analysis and internal friction.

Elongated grain structure was obtained in aluminium- and aluminium-calcium-deoxidized steels which were solution-treated at 1200°C, cold-reduced 40 to 83% and then annealed at 500 to 650°C. The results obtained may be summarized as follows:

(1) Slow heating for 4 hr from 400 to 600°C after cold reduction resulted in a faster decrease in hardness, and a coarser and more elongated grain structure than rapid heating to 600°C.

(2) In order to form elongated ferrite grains most AlN must be precipitated during annealing process, following solution treatment and cold reduction.

(3) It was found by the low frequency internal friction method that AlN precipitation occurred during the period from the recovery to the beginning of recrystallization under the condition of solution treatment at 1200°C, cold-reduction of 40% and 600 to 650°C anneal.

(4) Since aluminium-calcium-deoxidized steel showed lower (C+N) solubility in ferrite than aluminium-deoxidized steel, the stabilizing characteristics can be expected in the former steel.

Acknowledgment

The author expresses his gratitude to Prof. Dr. T. Ototani for advice and encouragement, to Prof. Dr. W. Köster for carefully reading the manuscript, and to Mr. Y. Takeyama for helpful chemical determination of AlN in steels.

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(18) W. Köster, L. Bangert und R. Hahn, Arch. Eisenhütt., **25** (1954), 569.

(19) K. Aoki, S. Sekino and T. Fujishima, Tetsu-to-Hagané, **48** (1962), 156.