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Effect of Annealing Cycle on Drawability and Microstructure of Cold Rolled Steel Sheet

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Abstract. The effect of the annealing cycle on microstructure, mechanical properties and draw ability of aluminium killed steel sheets at three different heating rates and holding times were investigated. Tensile test, hardness test, optical microscope and transmission electron microscope (TEM) techniques were utilized. The results of samples for different times in laboratory experiments were compared with the production samples. The results for these experiments showed that lower heating rates (12 °C/h) occurred at a lower annealing temperature, increase draw ability. This effect is attributable to the longer time available to initiate nucleation at the lowest heating rate. However, for all three heating rates (12 °C/h, 24 °C/h and 36 °C/h), the aluminium and nitrogen combine to form atmospheres or pre-precipitation clusters at polygonised sub-grains and as rolled boundaries, modifying the development of the recrystallized structure. Different amounts of AlN were precipitated prior to recrystallization for each heating rate resulting in different final recrystallized grain sizes, mechanical properties and drawabilities. The heating rate strongly influences recrystallization and drawability with a lower heating rate reducing both the temperature of the start of recrystallization and the recrystallized grain size. It is inferred that the lower rate promotes nucleation of recrystallization overgrowth of recrystallized. The effect of holding time on mechanical properties was to: (i) decrease tensile strength, yield strength and hardness and (ii) increase drawability, elongation and grain size. Tensile strength and hardness results from laboratory experiments were lower than those for production samples, but the trends were similar.

Keywords: Aluminium killed steel, mechanical properties, annealing, recrystallization, drawability.

1. Introduction

Low carbon aluminium killed steel is one of the most important products of the steel industry today. It had become important because it has many capabilities that no other commercial material can offer at low cost, properties such as relatively high drawability the ability to be readily formed at high production rates into various shapes, attractive surface finish, and good weldability. An important requirement for many applications involving sheet steel is, of course, good formability and the most common sheet forming method is press forming. The development of formability of low carbon aluminium killed steels depends on the alloying elements, impurities and inclusions in steel making, slab reheating furnace, finishing and coiling temperature in hot strip mill, total reduction, lubrication, heating rate, temperature and time annealing in cold rolling mill. All the process parameters above must be controlled to produce excellent deep drawing properties.



There are five major steps in the manufacturing of cold rolled and annealed sheet steels for extra deep drawing [1-9], as measured by the normal anisotropy (r-value). This property strongly controls the ability of the material to be deep-drawn into a flat-bottomed cup, and the strain hardening exponent (n-value) determines the ability of the material to be stretched. The first step is steel melting and casting, which includes control of elements in charging, such as copper, chromium, nickel, tungsten and eliminating as far as possible harmful elements such as sulphur. An increase in carbon content will decrease the r-value and excessive levels of impurities such as N₂, O₂, and inclusions including Al₂O₃ and SiO₂ will also decrease the r-value. Therefore, close composition control is required. The second step is hot rolling practice control. A high finishing temperature will increase the r-value for Al-killed steel because a high finishing temperature produces a more favourable annealed texture of cube-on corner rather more than a cube-on face. The most important step for aluminium killed steels is coiling practice, a lower coiling temperature leads to higher r-value because ALN is retained in solution in ferrite and can precipitate out on annealing after cold working with an increase in {111} grain orientation at the expense of {100} grain [10-15]. Therefore, the r-value becomes higher if the coiling temperature is low (<600°C). In the third step of cold rolling, the major variable is the reduction ratio, with the optimum r-value being obtained on annealing following a reduction ratio of 60% - 70%. The fourth step of batch annealing includes the rate of heating to the annealing temperature, the temperature and time of the hold. In aluminium killed steels the response to annealing varies with the aluminium content the r-value decreases monotonically with the heating rate in the case of low carbon rimmed steel, but as the Al content becomes higher, a peak r-value appears. The final step is an extension in the skin pass mill, which is important for stretching in the forming of cold rolled steel [16-20].

2. Experimental

The investigation was divided into two stages, the first stage consisted of a "mass production" investigation, including variations in reduction ratio in the cold rolling mill and the holding time during batch annealing. The second stage was the "laboratory scale simulation of industrial batch annealing" which allowed a closer study of the structure and properties of cold rolled and annealed samples, including effects of heating time, holding time and heating rate. These investigations in totality were used to obtain more detailed information about the mechanisms underlying the changes in the n and r-values with annealing conditions.

| ID | C (%) | Si (%) | Mn (%) | P (%) | S (%) | Al (%) | N (%) | Cr (%) | Ni (%) | Cu (%) | Nb (%) | V (%) | Mo (%) | Al/N |
|----|-------|--------|--------|-------|-------|--------|-------|--------|--------|--------|--------|-------|--------|------|
| A | 0.046 | 0.004 | 0.250 | 0.008 | 0.007 | 0.040 | 0.006 | 0.012 | 0.02 | 0.016 | - | 0.005 | 0.003 | 6.7 |
| B | 0.034 | 0.019 | 0.270 | 0.013 | 0.007 | 0.050 | 0.006 | 0.007 | 0.017 | 0.014 | 0.001 | 0.006 | 0.004 | 8.3 |
| C | 0.046 | 0.003 | 0.252 | 0.010 | 0.006 | 0.040 | 0.006 | 0.008 | 0.015 | 0.023 | 0.001 | 0.003 | 0.002 | 10 |
| D | 0.035 | 0.010 | 0.290 | 0.007 | 0.009 | 0.052 | 0.005 | 0.006 | 0.012 | 0.010 | 0.001 | 0.005 | 0.002 | 10.4 |

Table 1. Chemical compositions of steels used.

The normal anisotropy (r) of each annealed steel sheet was determined using a tensile specimen with a gauge length (l) and width (w) 50 and 25 mm, respectively. The r value was determined (similar to JIS No. 5) by stretching the sample by 15% and then using

measurements of the width in ten locations before (w_0) and after elongation ($w_{15\%}$) in Equation 1.

$$r = \frac{\varepsilon_w}{\varepsilon_h} = \frac{\ln\left(\frac{w_0}{w_{15\%}}\right)}{\ln\left(\frac{l w_0}{l w_{15\%}}\right)} \quad (1)$$

Tensile samples were taken 0° (L)° 45° (D) and 90° (T) relative to the rolling direction: An average value of r was determined from Equation 2.

$$r = \frac{(r_L + 2 \cdot r_D + r_T)}{4} \quad (2)$$

The strain hardening exponent, n , of each steel was calculated using the measured stress at 10% strain and the ultimate strength according to the Nelson-Winlock Table. The samples were examined using optical microscopy and transmission electron microscopy to detect precipitation of aluminium nitride and observation of the structures associated with recovery, recrystallization and grain growth. The mean grain size was measured using quantitative metallographic analysis according to ASTM E 112.

3. Results and Discussions

Effect of Annealing Cycle on Drawability. The results of mechanical tests for various holding times at 680 °C are shown in Figs.1 and 7. Figure 1 shows the effect of holding time on r -value and n -value. For holding at 680 °C from zero to 8 hours the r value increased, after which it decreased on holding time for up to 16 hours. The peak r -value occurred in 6 hours, for a heating rate of 24°C/h and the peak r -value was obtained in 4 hours for heating rate 36°C/h. From these data, it is evident that slow heating results in a higher r -value occurring at a longer holding time than for rapid heating. Figure 2 shows the effect holding time on n -value for the three heating rates. The trend in n value is similar to the trend in r -value above. For heating rates of 12, 24 and 36 °C/h, peaks in n -value occurred after 8 hours, 6 hours and 4 hours, respectively. The influence of grain size on r -value is significant during heating, with increasing grain size being associated with an increase in r and n value is shown in Figure 3. The grain size increases from 20 μm at 640 °C after 8 hours, to 35 μm after 20 hours. However, for holding time at 680 °C, the influence of grain size on r and n value is not significant.

The results of tensile tests for various holding times at 680 °C are shown in Figures 4 and 7. These curves show that the hardness, tensile and yield strength decrease with increasing holding time. It can be seen that an increase in the holding time increases the elongation, as a result of the increasing grain size. The ratio of tensile strength to yield strength gradually increased with increased holding time up to 20 hours. The effect of a high ratio of tensile to yield strength is to increase the exponent for strain hardening. While the strength properties decreased with increasing time for the laboratory samples studied, the drawability of the

experimental aluminium killed steel improved with increasing temperature or heating time to an optimal value corresponding to a particular holding time.

Comparing the r -values obtained from samples treated by the three different laboratory annealing cycles with the production scale. The curve of yield strength from the production samples was sharper than for laboratory samples, but both showed a decrease in the yield strength with time. Holding times that produced the coarsest grain sizes also resulted in lowest yield strength, tensile strength and hardness. Increasing the holding time, increased the r and n values, but the curves were not linear, with peaks being obtained at 4, 6 and 8 hours for heating rates of 12 °C/h, 24 °C/h and 36 °C/h, respectively. Figures 1 - 2 show there was no essential effect of holding on r and n -value from the actual production samples. The difference in the laboratory and production results is caused by differences in equipment and other practical factors. For example, the production scale treatment in a batch annealing furnace has many variables: coil stacking practice, furnace temperature, maximum permitted top outer edge temperature and annealing equipment (direct fired vs radiant-tube furnaces, fan size, convector plates). The annealing cycles and practices are compromise, in that the furnace temperature, firing time and permitted top outer temperatures are specified to sufficiently anneal the most difficult part of the coil to heat, the cold spot, which in current practice is approximately one third of the way into the bottom or middle coil from the eye of the coil.

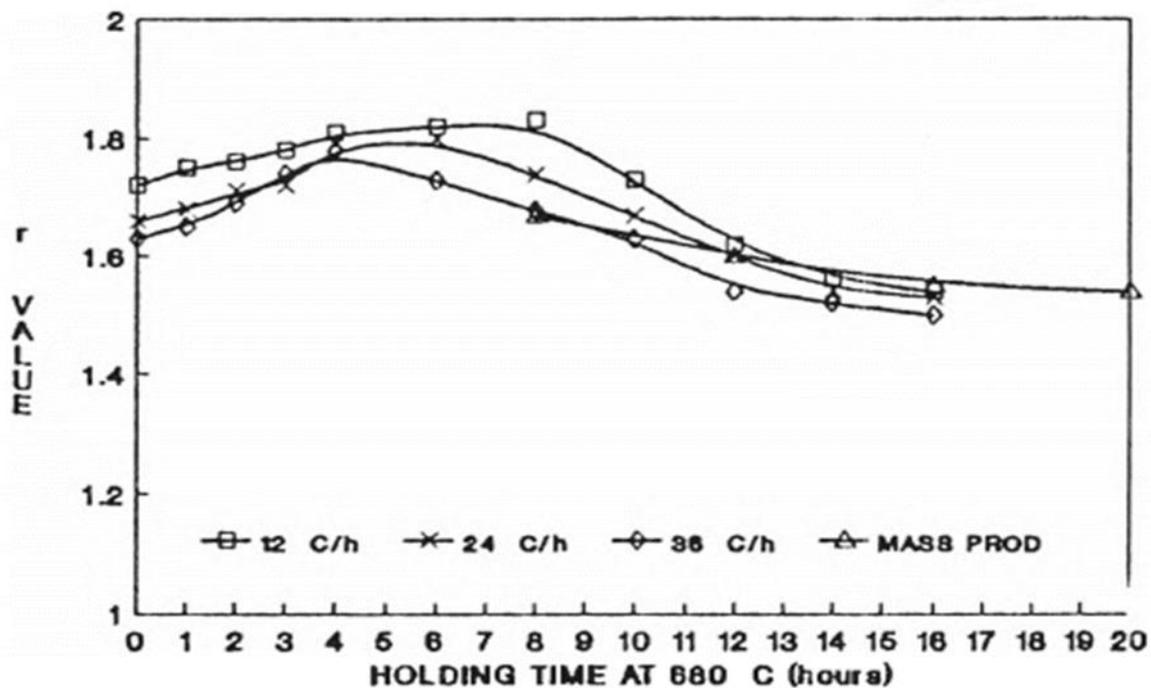


Figure 1. Effect of holding time on r value.

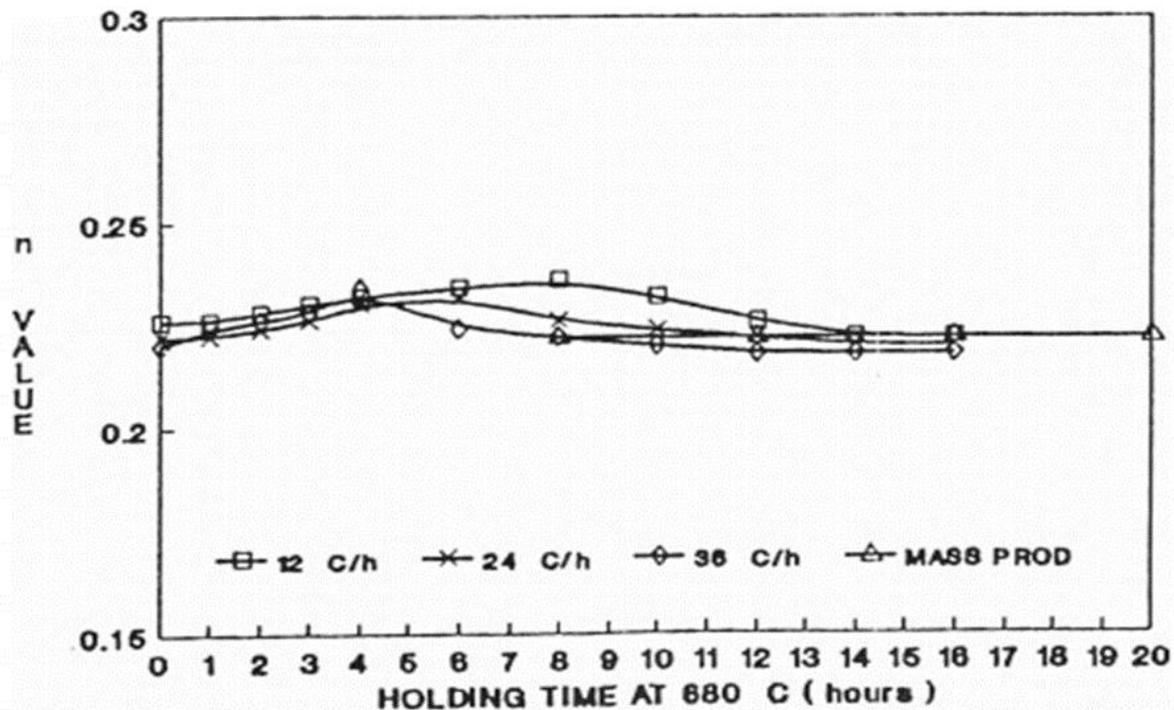


Figure 2. Effect of holding time on n value

Time at temperature during box annealing is considerably less important than maximum temperature attained. The difference in the laboratory and production results is also caused by extension in temper pass mill. Production scale is utilised extension in temper pass mill, but not used for laboratory scale. Temper pass is the term applied to the final processing of the cold rolled strip to impart the correct roughness of the surface and to strain sufficiently to suppress the yield point for the elimination of stretcher strains during forming treatment of the customer, so that the materials continue to stretch evenly up to the yield strength. An investigation by Shimizu et al [21] indicated that increasing the holding time and holding temperature resulted in a decrease in tensile strength of cold rolled rimmed steel sheet.

Pakkala et al [22] reported that very little improvement in properties is obtained by holding at subcritical temperatures for periods longer than 10 hours or in many instances beyond 5 hours. Furthermore, when annealing at 732 °C, no improvement is obtained from using time in excess of one hour. A rule of thumb that is frequently followed maintains that an increase in the temperature of 100 °C is roughly equivalent to a doubling of the time of annealing. Pakkala et al [22] studied the effect of annealing temperature and time on two nominally 0.040 inch thick rimmed steels of similar composition and processing (nominal 60 % cold reduction), except for coiling temperature. The results were generally representative of the trends expected for rimmed steel, i.e. the as-annealed hardness, yield, tensile strength and yield point elongation generally decreased as annealing temperature and annealing time are increased. Also, as would be expected, grain size increased with increases these variables. Elongation values unexpectedly showed little change with a variation of only 3 % and no consistent trend with the variables studied. The inconsistent relationship between elongation and the annealing variables may be related to local variations in composition and local variations in micro cleanliness.

Selected data from a study by Schwer et al [23] of the effects of annealing temperature on the as-annealed properties of low carbon rimmed steels (nominally containing 0.07 % C, 0.44 % Mn, 0.008 % P, 0.025 % S, 0.008 % Si, 0.02 % Cu, 0.01 % Ni and 0.004 % N.). indicated similar trends to those of Pakkala et al [22]. In addition, Schwer et al [23], showed an increase in the r- value and the n-value, associated with increasing annealing temperature. It should be noted that significant undesirable grain coarsening resulted from annealing these steels at 690 °C and higher, however, inasmuch as the annealing was conducted in stagnant air in a welded box, this grain coarsening could have been due in part to partial decarburisation. In this study, slow heating and cooling rates were used.

Espey [24] reported the results of his work with an 88 % cold reduction steel containing 0.09 % C, 0.45 % Mn, 0.007 %P, 0.026 %S, 0.003 %Si, 0.098 % Cu and 0.02 %Mo and related the mechanical properties to the effective temperature and location within a coil. Effective temperature is defined by Espey [24] as the temperature with slight upward variations at which the steel is held at for 3 hours. However, these result probably represent the most complete time/temperature work done on box annealing and are useful for predicting trends.

Trishman et al [25] conducted an extensive investigation of the effects of annealing temperature and time on the hardness and grain size of four low carbon steels. Their annealing practice was to heat at 482 °C/h to 538 °C, then at 1 °C/h from 482 to the annealing temperature, and furnace cooling after a specified hold at the annealing temperature at a rate that reportedly approximates plant cooling rates. These results indicate that increasing the holding time and holding temperature resulted in a decrease in hardness and an increase the grain size.

Thesima and Shimizu [26] reported on the effect of increasing the annealing temperature of rimmed steel. They concluded that whereas a temperature above Ac-1 may be good for drawability, on the basis of conical-cup values, inter-critical annealing may be deleterious to stretchability. Also, during annealing above the Ac1, cementite segregates to grain boundaries, and during cooling the precipitation of carbon is retarded, resulting in increased aging tendency due to carbon in solution.

Matsuda and Nakaoka [27] investigated the effect of increasing annealing temperature and cold reduction on the r-value and grain size of rimmed steel. Their data indicated that r-value is increased by increasing the annealing temperature from 700 °C to 800 °C. They further reported an almost linear relationship between ferrite grain growth and increase in r-value, which they expressed as:

$$r = r_0 + k^{-1/2} + d_0^{-1/2}$$

where:

k = cold reduction kept almost constant at 50-80 % ($k < 0$)

r = final average r-value

r_0 = average r-value after completion of of recrystallization.

d = average diameter (mm) of final ferrite grains

d_0 = average diameter of grains upon completion of recrystallization.

Matsuda and Nakaoka [27] suggested that this relationship is also applicable to low carbon Al-killed steel and extra low carbon Ti steels. Matsuda and Nakaoka [27] in the previously cited review paper reported the results of Takechi on the effects of increasing annealing temperature on the r-value of rimmed and killed steels. Takechi concluded that r-values of both rimmed and Al-killed steel increased as the annealing temperature is increased from about 600 °C-850 °C then decreased significantly when the annealing temperature was further increased. However, the increase in r-value for Al-killed steel appeared to be slight from about 732 °C-850 °C. Schwer et al [23] reported data for four heats of Al-killed steel properly processed on the hot mill and cold reduced 45 to 50 % before being annealed for 16 hours at temperatures ranging from 650 °C-746 °C. These results, which are the averages of data obtained for the four heats, indicated the average change in mechanical properties that can be expected for Al-killed steel sheet for an increasing annealing temperature. As expected, hardness, yield and tensile strengths decreased and r, n value, grain size and elongation increased with an increase in annealing temperature.

A study conducted at the USS Research laboratory [28] was concentrated primarily on the effects of cold reduction, but two annealing practices were included: 6 hours at 677 °C and 16 hours at 705 °C. The results of this study of sheet cold reduced 50, 60 and 70 %, showed a modest improvement in hardness, yield and tensile strength elongation, associated with the higher temperature, longer-time anneal. However, identical r-values and essentially identical r-values resulted from both annealing practices. These results also showed the expected increase in r-value and decrease in r-value with increasing cold reduction from 50 to 70 %. They did not show any deleterious effect of increasing cold reduction from 50 to 70 % on hardness, yield strength or tensile strength. Although the elongation values showed a decrease with increasing cold reduction, calculations indicate that this decrease is essentially due to the reduced thickness resulting from increased cold reduction, and is not an effect of cold reduction. Thesima and Shimizu [26] studied the effect of annealing temperature (690 °C-750 °C) and holding time (2 - 18 hours) on Erichsen, conical cup (CCV), and r-value. Their results, which are somewhat different for the two steels studied, generally showed a slight improvement in r-value with an increase in annealing temperature. For one steel, the Erichsen value (stretchability) decreased with increasing annealing temperature, whilst for the other, there was no consistent effect. Likewise, conical-cup values (drawability) improved with increasing temperature for one steel, but increased annealing temperature showed no consistent effect for other steel. Holding time increases from 2 to 18 hours showed no consistent effect on the properties.

Sardana [29] studied the effect of annealing at 700 °C (20 and 30 hours) vs annealing at 740 °C (23 and 30 hours). The latter temperature was in the two-phase or inter-critical region. Sardana's data suggest that whereas yield strength is generally reduced by increasing annealing temperature and holding time, increasing temperature and is always associated with improvements in elongation and cup test performance. In general, the references discussion tend to support the result obtained in the present work.

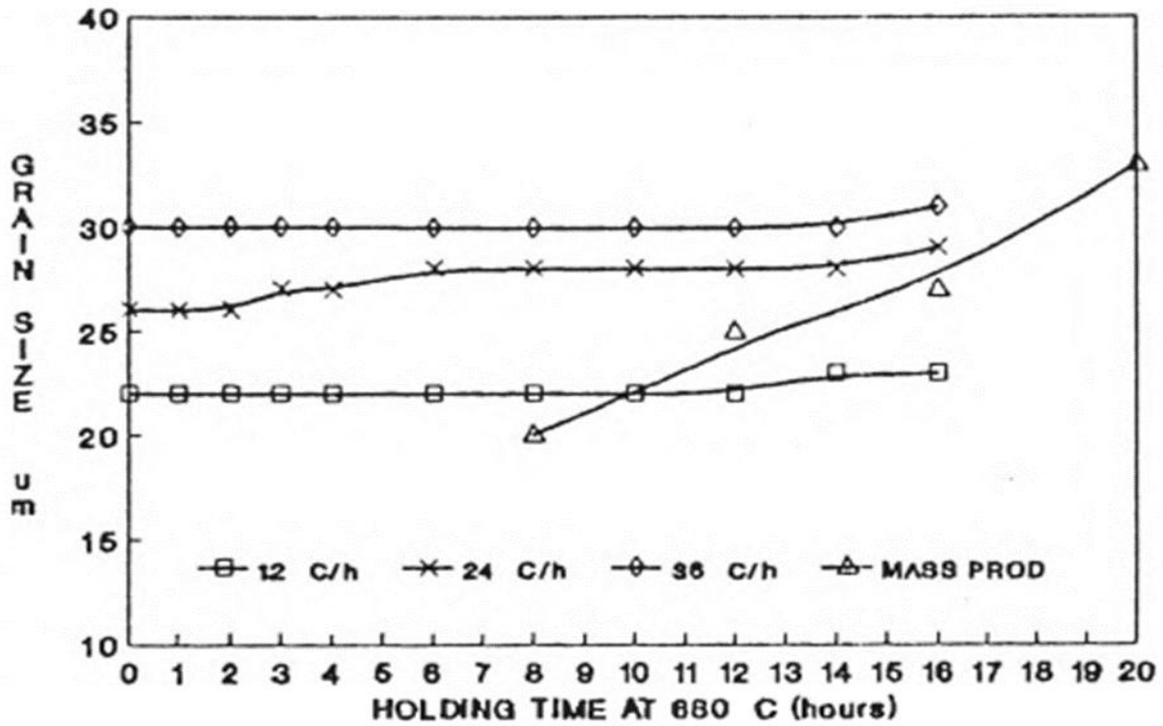


Figure 3. Effect of holding time on grain size

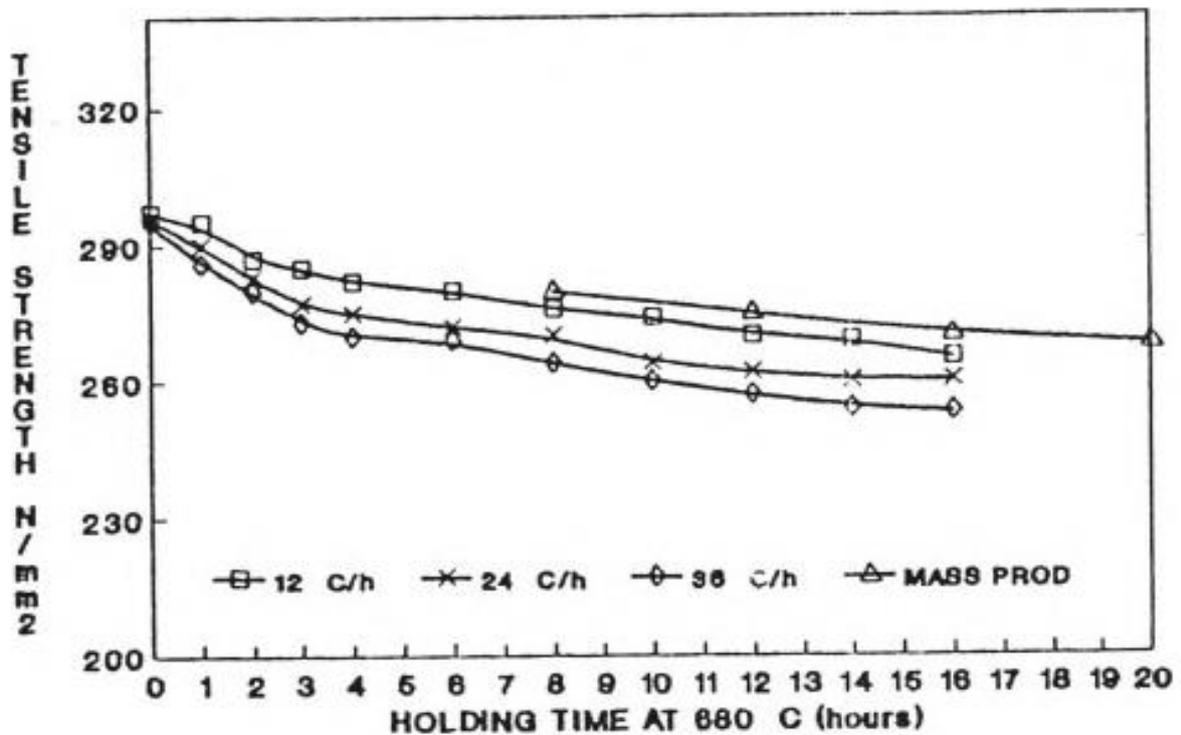


Figure 4. Effect of holding time on tensile strength.

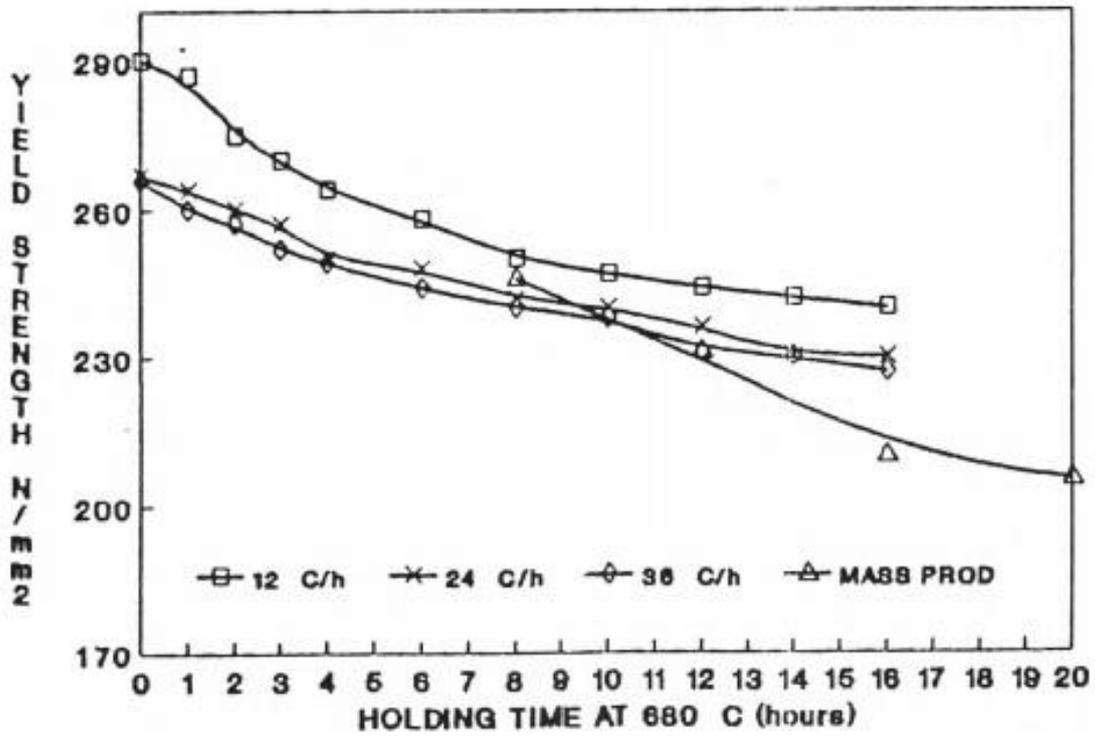


Figure 5. Effect of holding time on yield strength.

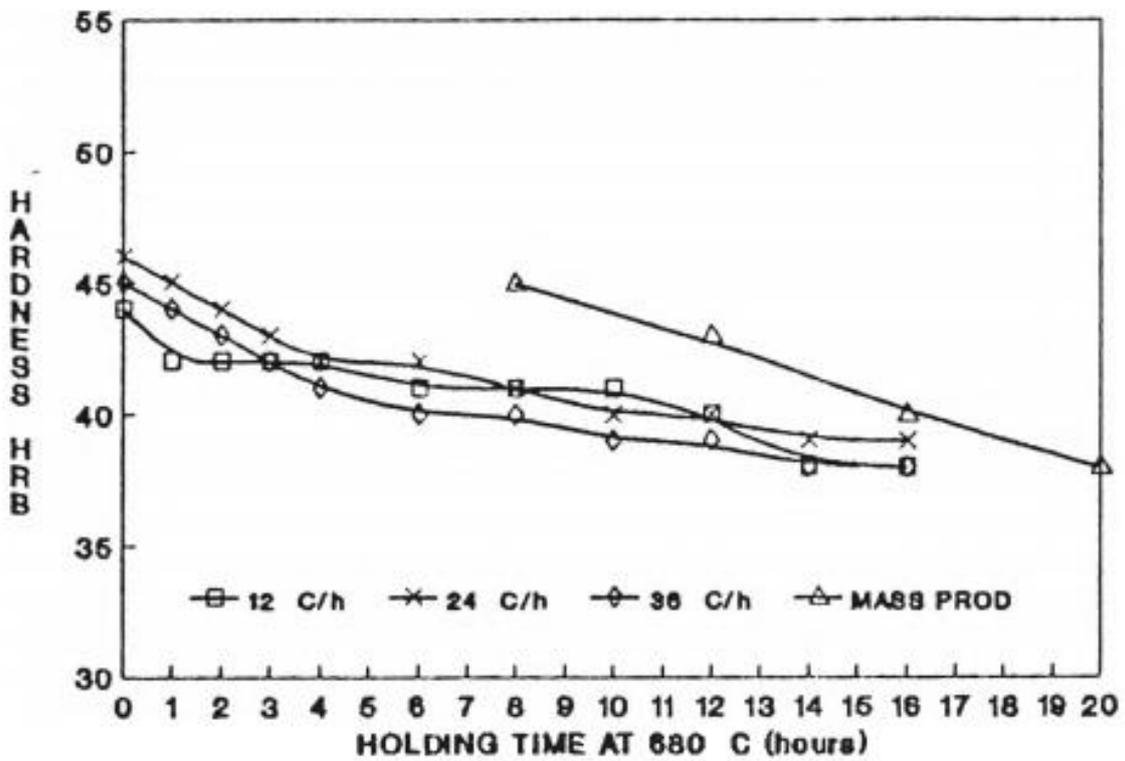


Figure 6. Effect of holding time on hardness.

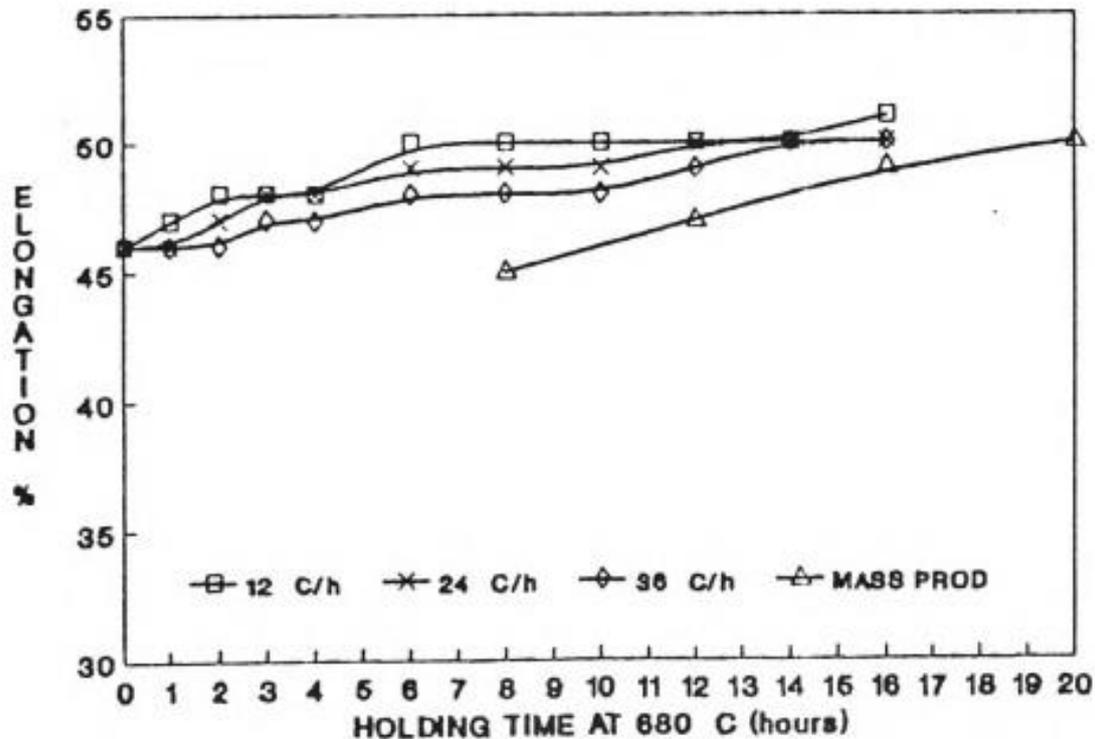


Figure 7. Effect of holding time on elongation

Effect of Annealing Cycle on Microstructure. Figure 8 shows the microstructure of mass-produced batch annealed steel. These photomicrographs show that the width of the grains is much smaller than the length of the grains. In other words, the structure is elongated or has a "pancake" shape. The effect of a holding temperature of 680 °C for times up to 16 hours is that grain size increases gradually, starting from a fully recrystallized grain size of approximately 20 μm and finishing at a size of about 35 μm after 20 hours. The grain dimensions after a holding time of 20 hours still produced a pancake shape.

The results obtained by metallographic point counting are shown as plots of volume fraction recrystallized versus time in Fig 9. The curves for different heating rates show the classic sigmoidal shape associated with recrystallization. There is an incubation stage, which depends on heating rate, followed by rapid recrystallization and a final stage during which recrystallization rate slows down as grain impingement occurs. A lower recrystallization temperature was associated with a lower heating rate. The recrystallized grain size of the steel used was determined for heating rates of 12 °C/h, 24 °C/h and 36 °C/h by the circular intercept procedure. The results are shown in Figs 3 and 10. The starting grain size as rolled grain condition was approximately 10 to 11 μm . For a heating rate of 12 °C/h recrystallization started at a temperature of 508 °C, when the grain size was approximately 13 μm . For a heating rate of 24 °C/h recrystallization started at 544 °C and for a heating rate of 36 °C/h the recrystallization started at temperature was 556 °C. The grain size at the start of recrystallization was similar in the three cases. During recrystallization, the grain growth at a heating rate of 36 °C/h was more pronounced than for a heating rate of 24 °C/h and also for 36 °C/h. The grain size averaged approximately 22 μm for a heating rate 12 °C/h, 26 μm for

rate strongly influences recrystallization. The smaller the heating rate the lower the temperature of the start of recrystallization and the smaller the recrystallized grain size. The implication heating rate 24 °C/h and 30 µm for 36 °C/h. From the grain size versus temperature curves, it is evident that the heating is that the lower rate promotes nucleation of recrystallization overgrowth of recrystallized grains. The effect of holding time on the recrystallized grain size at 680 °C is also presented in Fig 3. This plot shows that grain size mostly changed a little on holding and was lower at the lower heating rate. The grain size for heating rate 12 °C/h was approximately constant at 22 - 23 µm, for 24 °C/h, 26 to 29 µm, and for 36 °C/h 30 to 31 µm.

Optical microscopy also identified features associated with the mode of nucleation of recrystallization. From observations of the grain boundaries during the course of recrystallization, it was evident that there was a large degree of grain boundary migration occurs as a precursor to recrystallization. Fig 11 an extraction replica of this study shows that the AlN has precipitated at the former as rolled grain and sub-grain boundaries by the time recrystallization are substantially complete. It presented that the recrystallized grains are elongated. Inspection with TEM of the areas within these grains reveals traces of aluminium nitride precipitates at the former as rolled grain boundaries, the results for all three heating rates indicated that the grain size is relatively constant with increasing time up to 16 hours at a holding temperature of 680 °C, because of retardation of grain motion by layer of precipitates of AlN at prior cold worked boundaries. Dunne and Dunlea [16] concluded that in aluminium killed steel, nucleation by sub-grain growth is selectively inhibited by clusters or precipitates at sub-grain boundaries and growth of recrystallized grains is anisotropically retarded by precipitates at prior cold worked boundaries. They concluded that texture development is primarily dependent on nucleation events and that grain shape and growth are determined by other physical conditions. Elongated grain is frequently associated with a favourable texture, but in some cases, the link between texture and elongated grains can be broken. By increasing the time and temperature of pre-recrystallization anneal, and hence the amount of AlN precipitated.

Goodenow, Michalak and Leslie [9-11] observed that elongation of the recrystallized grains increased. Precipitation of AlN occurred throughout the annealing process and the extent of precipitation was determined by standard analytical techniques, which are said to account only for precipitate particles greater than about 200 Å.

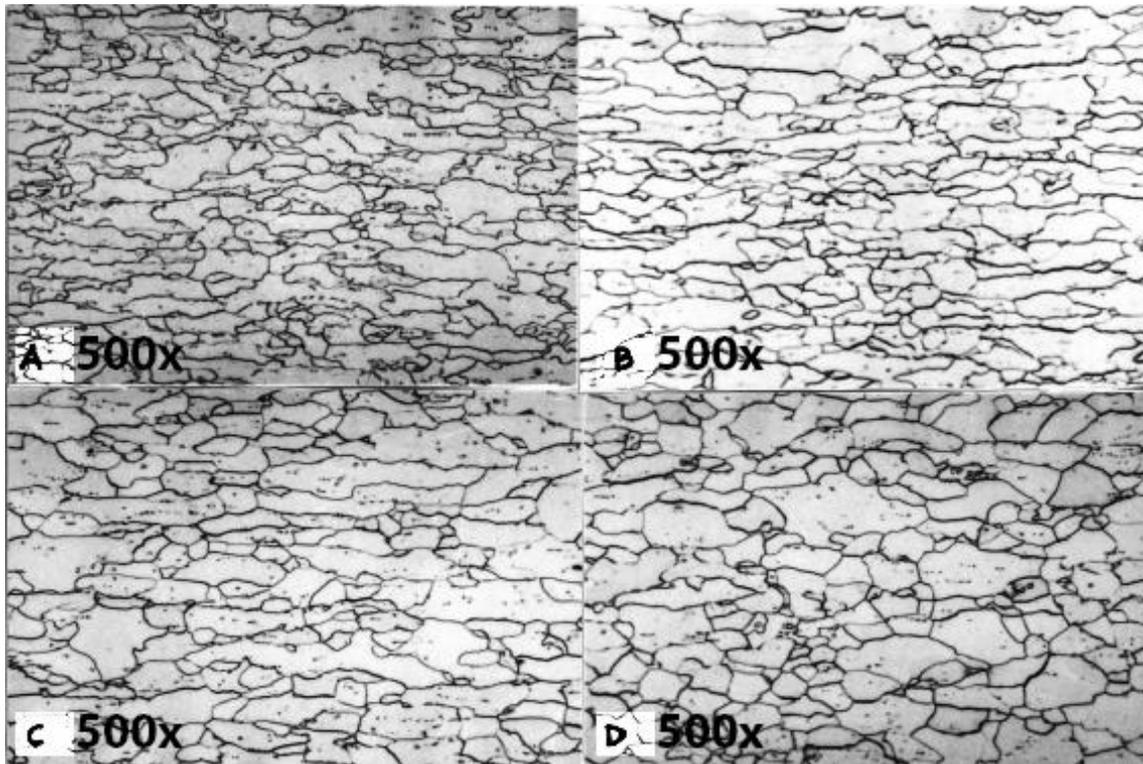


Figure 8. Photomicrographs (500 X) of Al-killed steel fully recrystallized after holding at (a) 640 °C for 8 hours (b) 680 °C for 12 hours (c) 680 °C for 16 hours (d) 680 °C for 20 hours.

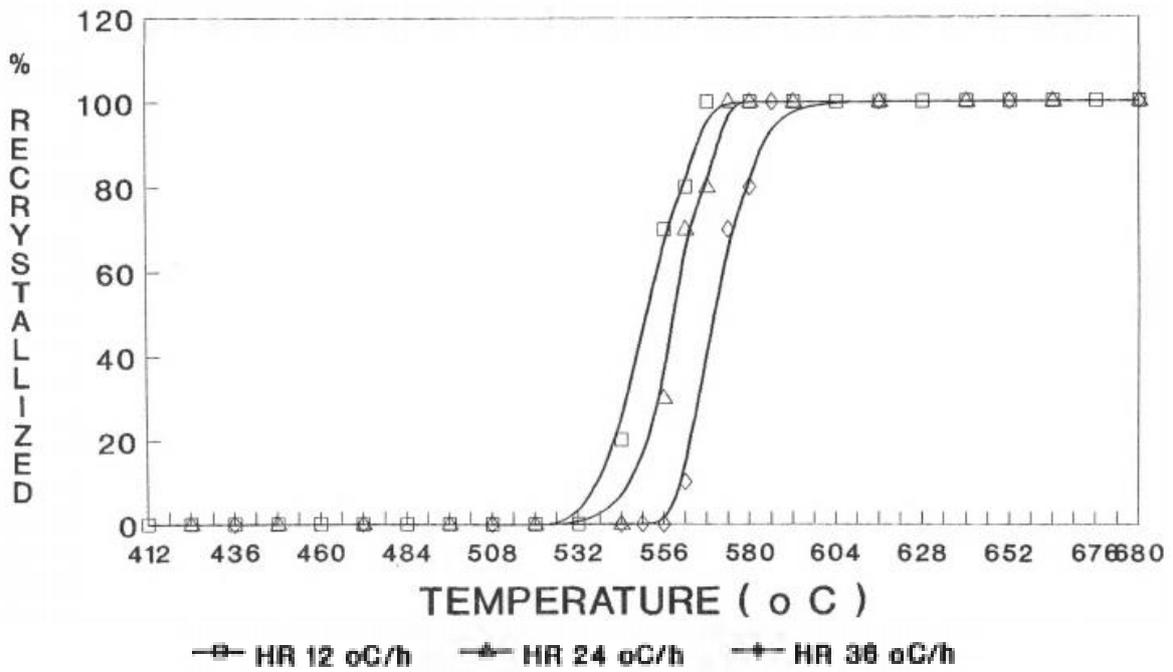


Figure 9. The volume fraction of recrystallized grain as a function of temperature.

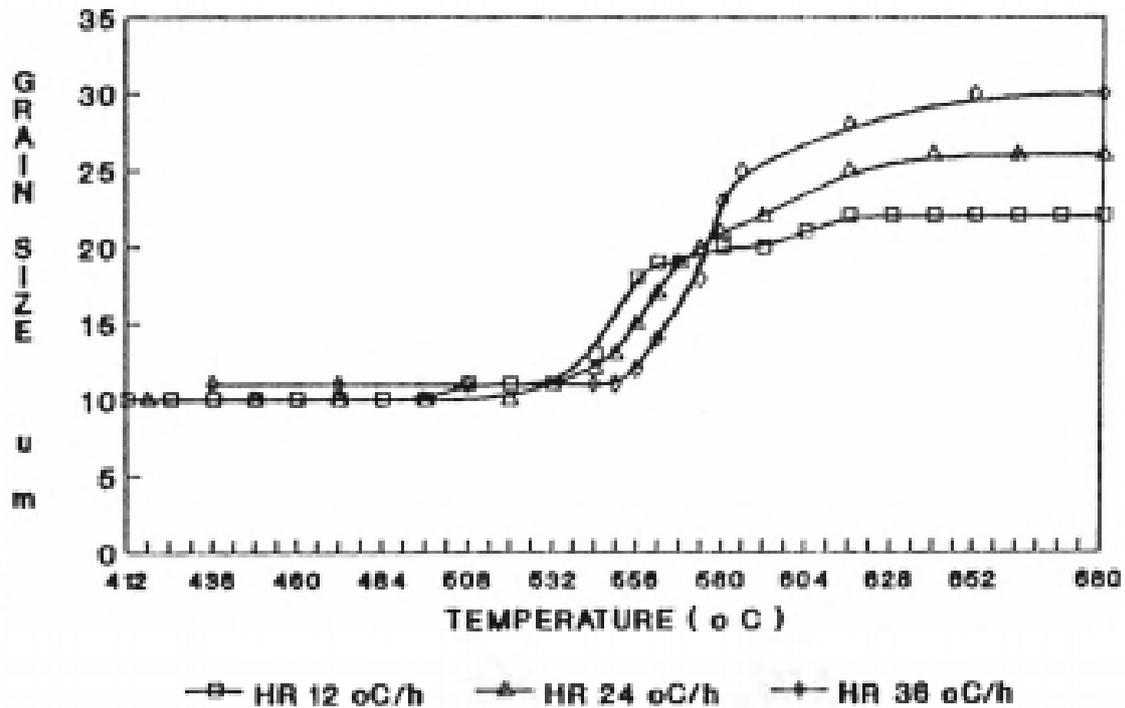


Figure 10. Effect of temperature on grain size

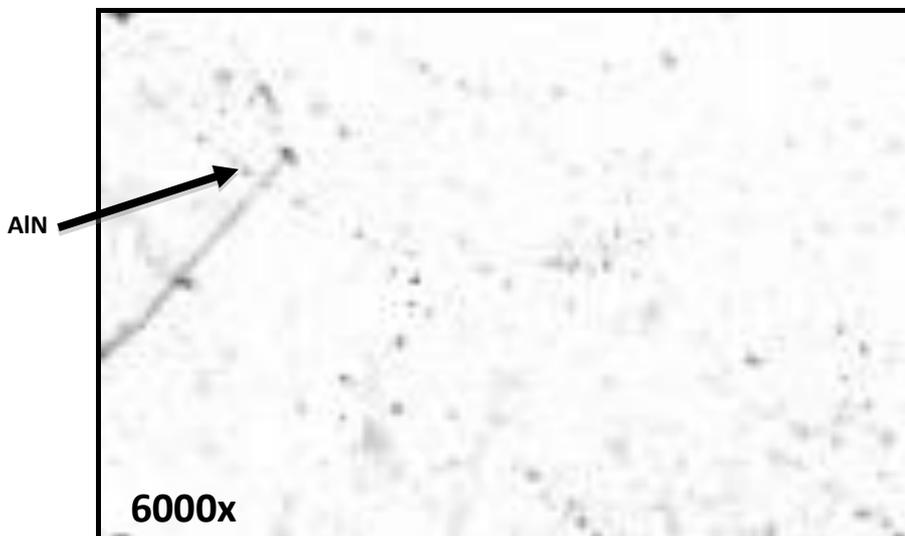


Figure 11. Carbon extraction replica by transmission electron microscope (TEM) of aluminium killed steel annealed by holding time at 680 °C for 16 hours, with the heating rate of 24 °C/h: Aluminium nitride precipitate delineates cold worked grain and sub-grain boundaries.

4. Conclusions

The low carbon aluminium killed steels studied in this work have the r-value increased gradually as the temperature increased for the heating rate of 12°C/h, and for isothermal holding at 680°C, the r-value increased for up to 8 hours, after which it is decreased. Likewise, for the heating rate of 24°C/h the r-value increased to a peak after 6 hours, and for the heating rate of 36°C/h the peak occurred after 4 hours. It is caused by the aluminium and nitrogen combine to form atmospheres or pre-precipitation cluster at polygonized sub-grain and as rolled boundaries, modifying the development of the recrystallized structure to elongated, nucleation by sub-grain growth is selectively inhibited by clusters or precipitates at sub-grain boundaries. However, after holding time of 8, 6, 4 hours for the heating rate of 12, 24, 36 °C/h, AlN precipitate cannot lock the elongated structure anymore.

The effect of holding time on mechanical properties was: (i) decrease in tensile strength, yield strength and hardness; (ii) increase in elongation and grain size. Tensile strength and hardness results from laboratory experiments were lower than those for production samples, but the trends of curves were similar. Holding times that produced the coarsest grain size also resulted in the lowest yield strength, tensile and hardness, for all three heating rates.

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