

Effect of sulphur content on the
recrystallisation behaviour of cold worked low
carbon Aluminium-killed strip steels

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recrystallisation behaviour of cold worked
low carbon Aluminium-killed strip steels

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The influence of the sulphur and nitrogen content on the static recrystallisation behaviour of cold worked low carbon Al-killed strip steels was investigated. This was in response to the observation made by some users of these steels that the recrystallisation process after cold work was “sluggish” in some steels and, therefore, this was affecting their productivity as the continuous annealing lines had to be run slower and the batch annealing cycles required a higher annealing temperature and thus more energy input.

Two groups of Al-killed low carbon strip steels, one with low (<10 ppm) and the other with medium to high (> 70 ppm) sulphur content were studied. It was found that sulphur had an indirect but significant effect on the recrystallisation behaviour after cold work of these steels. In the high sulphur content steels, the

sulphur precipitated as manganese sulphide (MnS) and copper sulphide (CuS/Cu₂S) coarse particles. These sulphide particles, particularly MnS, were the favoured nucleation sites for aluminium nitride (AlN) and the heterogeneous nucleation encouraged the precipitation of AlN during coiling after hot rolling. The result was that the AlN in medium to high sulphur content steels was generally associated with coarse sulphides and, therefore, the mean particle size of the AlN/MnS particles in the as-coiled steel prior to cold working and annealing, was generally much coarser than in the steels with low sulphur content. In these low sulphur content steels, the AlN nucleated homogeneously in the matrix or heterogeneously on grain boundaries or dislocations during coiling. Consequently the mean particle size of AlN in these low sulphur content steels was significantly finer, often less than 30 nm in diameter and, therefore, these particles were more effective in retarding the recrystallisation process through Zener-pinning of dislocations and moving recrystallisation fronts.

The effect of lower sulphur content was exacerbated by lower coiling temperatures (~600 °C) i.e. the recrystallisation start time increased with a decrease in coiling temperature as the AlN particles remained small due to a low coarsening rate. On the contrary, no significant sensitivity to coiling temperature was observed in the time for the start of recrystallisation after cold work in medium to high sulphur steels within the coiling temperature range 600 to 650 °C. The conclusion was that sulphur, in the presence of manganese, does not hinder

recrystallisation after cold work in Al-killed steels but promotes heterogeneous nucleation and growth of AlN on the coarser MnS. The effect is that the effective mean particle size of the AlN becomes much larger as it is not isolated homogeneously as in low sulphur steels but is tied to the sulphides. The sulphur (ppm) dependent empirical expressions for the recrystallisation start times $t_{5\%}$ at isothermal annealing temperature of 610 °C for steels coiled at temperatures of 600 and 650 °C were found to be:

$$t_{5\%} = 33.78\exp(-0.0345S) \text{ for } 600 \text{ }^\circ\text{C and}$$

$$t_{5\%} = 0.99\exp(-0.008S) \text{ for } 650 \text{ }^\circ\text{C.}$$

Recrystallisation arrest was observed during the annealing process after cold work of as-quenched specimens. The apparent activation energy of the process that led to the recrystallisation arrest, being 230 kJ mol⁻¹, is of the order of the activation energy for the diffusion of aluminium in ferrite, i.e. 196.5 kJ mol⁻¹. Since the precipitation process of AlN is controlled by the slower diffusion of aluminium, this was an indication of the nucleation/clustering of fine particles of AlN, which consequently halted the recrystallisation process through effective Zener drag. This Zener-pinning effect was used to estimate indirectly the precipitation start and finish times for the AlN during the isothermal annealing after cold work of these steels.

The solubility of AlN in austenite has mostly been studied by others using the Beeghly selective dissolution technique which has often been criticised for its limitations in its lack of sensitivity to the presence of very fine AlN particles, i.e. < 10 nm, and its failure to separate the AlN from other nitrides. In this study the thermoelectric power technique [TEP] was used to study the solubility of AlN in austenite in commercial low carbon Al-killed steels; one group with a medium to higher sulphur content and the other with a lower one. The equilibrium solubility equation thus obtained was determined as:

$$\text{Log}[\%Al][\%N] = 2.6 - \frac{9710}{T}$$

where the aluminium and the nitrogen contents are in weight percentages and T is the absolute solution temperature in Kelvin.

It was found that the AlN solubility equation derived here predicted slightly higher solubility temperatures for the AlN if compared to those derived from the Beeghly method. It was also confirmed that the equilibrium solubility of AlN was not sensitive to the sulphur content (unlike the precipitation behaviour), which is in agreement with the results from others obtained through the Beeghly technique.

Key words: Heterogeneous and homogeneous nucleation, Zener-pinning force, mean particle size, activation energy, driving force, static recrystallisation, Avrami exponent and constant.

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rolled in 4 passes and then coiled at 600 °C for 1 hour; TEP1 = absolute TEP value immediately after coiling while TEP2 = absolute TEP value after annealing at 800 °C for 2 hours cooled to 600 °C and annealed for 10 minutes and quenched in water. Error = 0.033 $\mu\text{V K}^{-1}$.

Table 13.1: TEP coefficients K_{AIN} for AlN obtained from the five steels that were studied and compared to published values.

Table 13.2: Parameters that have been used to calculate the activation energy for the isothermal and homogeneous nucleation of AlN in ferrite ΔG^*_{AIN} .

Table 13.3: Estimated volume fraction V_v of the precipitated AlN during isothermal annealing at 610 °C in 70 percent cold worked steels HS140-104, LS70-38, LS2-65 and HS90-12.

Table 13.4: Isothermal annealing temperatures and times, the modelled particle radii $r_{(t)}$ and their corresponding modelled Zener drag force P_z in J m^{-3} at the point when the recrystallisation resumes after the arrest.

Table 13.5: Parameter values for the calculations of ΔG_v , ΔG^* and r^* for the homogeneous nucleation of MnS, AlN and Cu_2S .