# VANADIUM IN INTERSTITIAL-FREE STEELS

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## ABSTRACT

The possibility of using vanadium in interstitial free steels has been examined. Vanadium carbo-nitride is less stable than other microalloy carbo-nitrides but conditions have been defined which will give high levels of plastic anisotropy. The interstitial free steels microalloyed with vanadium show lower recrystallisation temperatures than do other microalloyed steels.

## 1. INTRODUCTION

Many of the modern strip steels are manufactured to high levels of purity and cleanliness which enhances the formability of strip; almost all of these are manufactured using continuous casting. The formability of the steels can be enhanced further by making them "interstitial free". Interstitial free steels are characterised by low strength levels with excellent formability, especially if texture is controlled to give reasonably high plastic anisotropy ($r_m$) values. These steels have been made using either titanium or titanium-niobium additions. The niobium addition has resulted in severe retardation of recrystallisation during continuous annealing, requiring very protracted times or high annealing temperatures. Such treatments often lead to distortion of the strip during the continuous annealing cycle. Titanium additions sufficient to stabilise the interstitial elements can cause problems during galvanising, whilst lower titanium levels, augmented by niobium cause problems with recrystallisation as outlined above.

It has been suggested that a vanadium treated steels might exhibit the interstitial free characteristics without such a drastic retardation of recrystallisation. This paper details a programme of work comparing the recrystallisation kinetics and residual mechanical properties of aluminium, vanadium, titanium, titanium-vanadium and titanium-niobium treated steels.

## 2. BACKGROUND

The effects of microalloying additions of niobium, vanadium and aluminium on the recrystallisation of cold worked 0.15% C steels have been studied previously [1,2]. It was found that the fine interphase precipitates capable of precipitation hardening produced a significant retardation of the recrystallisation process. The particle size in these materials were of the order of 2-4nm in diameter. Steels in which the particles had been previously coarsened to 20-30nm in diameter showed similar recrystallisation kinetics to those of plain carbon steel. These results have been interpreted in terms of a Zener particle pinning mechanism, where the finer particles are capable of pinning the subgrain boundaries, whereas the larger particles, although capable of inhibition of grain growth with 20μm diameter (or larger) grains, cannot inhibit subgrain growth [3] with subgrains of 0.5-2.0μm in diameter.

Recent work [4], has shown that precipitates which are confined to grain boundaries or subgrain boundaries can be ten times more efficient than randomly distributed ones, which would account for the texture controlling mechanisms in aluminium treated steels. Control of the precipitation is therefore vital in controlling recrystallisation. Rapid heating of aluminium treated steels may allow recrystallisation to precede precipitation [5]. Microalloyed interstitial free steels, provided that precipitation can be encouraged to give coarse particles, should recrystallise reasonably quickly. The earlier results [1,2] suggest that recrystallisation would suffer retardation if the particle size and distribution were such as to give significant dispersion strengthening, and that recrystallisation would only occur if the particles coarsened sufficiently to eliminate any significant strengthening. It should be pointed out that the vanadium additions gave less retardation of recrystallisation than was produced by niobium additions [1]. This merely reflects the more rapid coarsening of vanadium carbo-nitrides compared with niobium carbo-nitrides.

## 3. EXPERIMENTAL PROCEDURE

A series of experimental low carbon steels were manufactured and are shown in Table I. The vacuum melted steels were cast into 50kg ingots which were machined for surface finish and subsequently forged to 100x50mm flats. The flats were hot rolled to 1200x110x3.5mm strip in two heats, finishing at 950°C, and then furnace cooled.

The hot rolled steels were pickled at 50-55°C in a 20% by volume sulphuric acid bath until the surface oxide scale was removed and then washed and dried. The pickled steels were subsequently 70% cold rolled using a Marshall Richards rolling mill in 2 high configuration.

**Table I: Chemical analysis of steels 1-7 (wt%)**

| Steel No. | C | Si | Mn | S | P | Ti | Nb | V | Al | O | N |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0.004 | <0.02 | 0.26 | 0.005 | 0.005 | - | - | - | 0.049 | 0.0073 | 0.0025 |
| 2 | 0.007 | 0.02 | 0.23 | 0.004 | <0.005 | - | - | 0.042 | 0.046 | 0.0033 | 0.0024 |
| 3 | 0.005 | <0.02 | 0.21 | 0.004 | <0.005 | - | - | 0.086 | 0.017 | 0.0040 | 0.0026 |
| 4 | 0.003 | <0.02 | 0.21 | 0.004 | <0.005 | 0.056 | - | <0.005 | 0.029 | 0.0026 | 0.0022 |
| 5 | 0.004 | <0.02 | 0.21 | 0.003 | <0.005 | 0.023 | - | 0.041 | 0.047 | 0.0024 | 0.0020 |
| 6 | 0.007 | 0.01 | 0.21 | 0.003 | 0.005 | 0.019 | - | 0.080 | 0.028 | 0.0066 | 0.0023 |
| 7 | 0.007 | <0.02 | 0.21 | 0.003 | <0.005 | 0.022 | 0.021 | <0.005 | 0.042 | 0.0033 | 0.0028 |

### 3.1. Recrystallisation studies

The cold rolled steels were cut into specimens oriented transverse to the rolling direction. Continuous annealing simulation recrystallisation studies were carried out covering the temperature range 600-800°C, and times from 10-600s in order to quantify the extent of recrystallisation. Hardness measurements using a Vickers pyramidal diamond indenter were taken of the 70% cold rolled samples, at a load of 20kg, and of the heat treated material, at a load of 10kg. A type K thermocouple spot welded to the specimens and linked to a chart recorder gave plots of the temperature against time for the heat treatment. The temperatures and times were calculated from the plots and the Larson-Miller parameter [6], P, for the heat treated steels calculated from Equation 1.

$$P = T\,(20 + \log t\,) \qquad (1)$$

where T is the temperature in K and t is the time in hours. Plots of hardness against P were produced.

### 3.2. Mechanical Properties

Tensile test pieces were manufactured to BS EN 10 002-1:1990, for non proportional test pieces [7], from the hot rolled and 70% cold rolled steels in three orientations, parallel, transverse, and at 45° to the rolling direction . The 70% cold rolled tensile test pieces were heat treated for 30s at 800°C to simulate continuous annealing. The tensile test pieces were surface ground with 1200 grit SiC papers to ensure the removal of any stress raisers and tested using a Mayes tensile testing machine. The hot rolled steels were tested at a crosshead separation rate of 0.625mm/minute and the heat treated steels were tested at a rate of 1.0mm/minute. During the tensile test the anisotropy ratio was measured at 10% strain. The plastic strain ratio, r, of width to thickness in a sheet is shown in Equation 2 [6].

$$r = \frac{\ln\ w_o/\,w}{\ln\ h_o/\,h} \qquad (2)$$

where $w_o$ and w are the initial and final width and $h_0$ and h are the initial and final thickness. Precise measurements of thickness are difficult to make on thin sheets, using the constancy of volume relationship Equation 2 can be rewritten as shown in Equation 3. The widths of the tensile test pieces were measured using a digital micrometer and the value of r calculated from Equation 3.

$$r = \frac{\ln\ w_o/w}{\ln\ \dfrac{wL}{w_o L_o}} \qquad (3)$$

where $w_o$ and w are the initial and final width and $L_o$ and L are the initial and final gauge lengths. The average anisotropy ratio $r_m$ was calculated from Equation 4.

$$r_m = \frac{r_0 + 2r_{45} + r_{90}}{4} \qquad (4)$$

where $r_0$, $r_{90}$ and $r_{45}$ are the r values calculated parallel, transverse and at 45° to the rolling direction respectively.

Plots of applied load against extension were obtained from the chart recorder. Values of lower yield stress (LYP) or 0.2% proof stress (0.2%PS), yield point elongation (YP El) and ultimate tensile strength (UTS) were calculated from these plots. A log-log plot of the true stress-strain curve in the region of uniform plastic deformation, up to maximum load, was used to obtain values of the strain hardening exponent, n.

3.3. Microscopy and Texture

Metallurgical specimens were prepared and examined. To investigate the formation of annealing textures 25mm diameter discs were punched from 70% cold reduced and annealed steels and (200), (220) and (222) pole figures were obtained.

3.4. Bake Hardening

Tensile test piece blanks of 70% cold rolled steels 2,3,5 and 6 oriented parallel to the rolling direction were heat treated at 800°C for 30s and then temper rolled 2%. The temper rolled blanks were immediately manufactured into tensile test pieces as for samples above and then baked hardened at 170°C for 20 minutes. The mechanical properties of temper rolled and bake hardened steels were obtained as for the steels stated above.

3.5. Batch Annealing

Tensile test piece blanks of 70% cold rolled steels 2,3,5 and 6 oriented parallel to the rolling directions were batch annealed as shown in Figure. 1. The batch annealed steels were then temper rolled 2% and bake hardened as above. The mechanical properties of the batch annealed steels were obtained for the steels stated above.

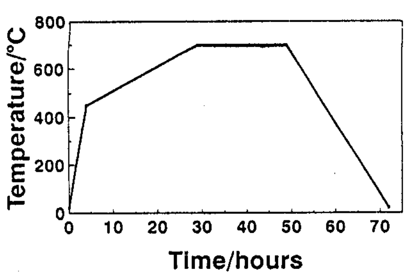


**Figure 1. Batch Annealing Cycle**

## 4. RESULTS

A plot of hardness (Hv) against Larson-Miller (L-M) parameter for steel 2, typical of those obtained for steels 1-7, is shown in Figure 2. The recrystallization data of steels 1-7 calculated from the L-M parameter plots are shown in Table II and the L-M parameters for recrystallization increase from steel 1 to 7. Vanadium

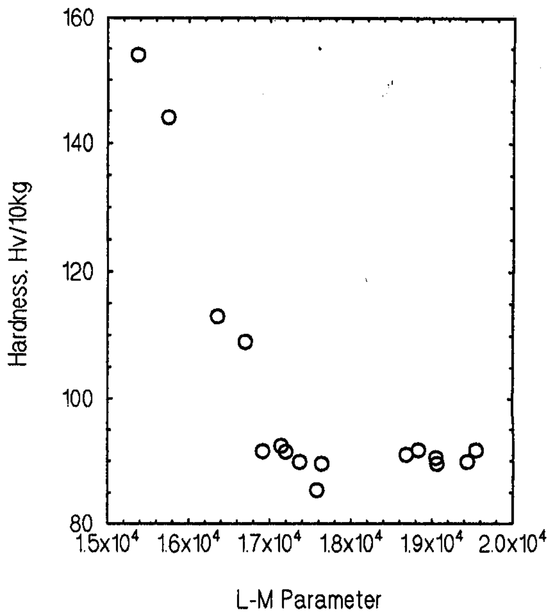


Figure 2. Recrystallisation behaviour of steel 2.

**Table II : Recrystallization data for steels 1-7.**

| Steel No. | L-M Par for Rec/$10^4$ | T for 30s/°C | T for 60s/°C | t for 700°C/s | t for 750°C/s |
|---|---|---|---|---|---|
| 1 | 1.66 | 656 | 640 | 4.6 | 0.7 |
| 2 | 1.68 | 667 | 651 | 7.3 | 1.0 |
| 3 | 1.70 | 677 | 662 | 11.5 | 1.6 |
| 4 | 1.76 | 709 | 693 | 44.1 | 5.8 |
| 5 | 1.77 | 714 | 698 | 54.6 | 7.1 |
| 6 | 1.78 | 720 | 704 | 70.8 | 9.0 |
| 7 | 1.86 | 765 | 748 | 470 | 54.7 |

additions (steels 2 and 3) increase the recrystallisation parameter, but not to the same extent as the titanium additions (steel 4). Vanadium additions to a titanium steel (steels 5 and 6) further increase the recrystallisation parameter but not to the same extent as the titanium-niobium steel (steel 7).

The tensile test data for steels 1-7 are shown in Table III. For the hot rolled steels, samples showing discontinuous yielding had higher yield points compared with those showing continuous yielding behaviour, but had comparable ultimate tensile strengths; yield point elongations were in the range 3-5%. Steel 1 is a plain carbon steel and therefore would be expected to show discontinuous yielding behaviour. The other steels showed mixed discontinuous and continuous yielding, and their occurrence was random within a single steel from one orientation in one hot rolled strip to another and it was

**Table III : Tensile test data for hot rolled and 70% cold rolled heat treated steels 1-7.**

| Process | Steel No. | Elong/% | LYP/MPa | 0.2%PS/MPa | UTS/MPa | YP El/% | n |
|---|---|---|---|---|---|---|---|
| Hot Rolled | 1 | 50 | 168 | - | 273 | 5 | 0.31 |
| | 2 | 49 | 183 | - | 262 | 5 | 0.31 |
| | 3 | 47 | 185 | - | 269 | 5 | 0.29 |
| | 4 | 53 | 135 | 86* | 245 | 3 | 0.32 |
| | 5 | 52 | 147 | 96* | 254 | 3 | 0.31 |
| | 6 | 49 | 180 | - | 270 | 4 | 0.30 |
| | 7 | 50 | 148 | 95* | 261 | 5 | 0.31 |
| 70% Cold Rolled & Heat Treated 800°C for 30s | 1 | 41 | 203 | - | 276 | 4 | 0.22 |
| | 2 | 43 | 216 | - | 299 | 4 | 0.30 |
| | 3 | 41 | 243 | - | 310 | 6 | 0.27 |
| | 4 | 45 | - | 135 | 294 | - | 0.33 |
| | 5 | 42 | 215 | - | 299 | 4 | 0.28 |
| | 6 | 43 | 239 | - | 314 | 6 | 0.30 |
| | 7 | 42 | - | 137 | 318 | - | 0.32 |
| 70% Cold Rolled, Heat Treated 800°C for 30s, Temper Rolled 2% | 2 | 40 | - | 243 | 339 | - | 0.17 |
| | 3 | 39 | - | 241 | 347 | - | 0.17 |
| | 5 | 45 | - | 207 | 317 | - | 0.23 |
| | 6 | 36 | - | 221 | 337 | - | 0.28 |
| 70% Cold Rolled, Heat Treated 800°C for 30s, Temper Rolled 2%, Bake Hardened 170°C for 20 mins | 2 | 30 | 293 | - | 347 | 4 | 0.19 |
| | 3 | 36 | 295 | - | 364 | 3 | 0.18 |
| | 5 | 42 | - | 191 | 318 | - | 0.24 |
| | 6 | 40 | 216 | - | 339 | - | 0.26 |
| 70% Cold Rolled, Batch Annealed | 2 | 47 | - | 108 | 298 | - | 0.37 |
| | 3 | 49 | - | 109 | 299 | - | 0.38 |
| | 5 | 43 | - | 117 | 300 | - | 0.35 |
| | 6 | 37 | - | 108 | 287 | - | 0.41 |

* - continuous yielding.

more usual to see a yield point.

Cold rolled and annealed steels 1,2,3,5 and 6 show discontinuous yielding behaviour (V and V-Ti alloys), steels 4 and 7 show continuous yielding behaviour (Ti and Ti-Nb alloys).

The recrystallised steels again had higher yield points and ultimate tensile strengths than the hot rolled steels. Hardness values were of the same order as the hot rolled steels, ie approximately 100 $Hv_{10}$ less than the 70% cold rolled steels. Temper rolling removed the yield point showing continuous yielding and decreased the yield stress. After bake hardening the yield point returned for steels 2 and 3 with an increase of 50 MPa and 54 MPa respectively. For steels 5 and 6 the return of the yield point was not clearly defined.

The batch annealed steels all showed continuous yielding indicating the complete removal of interstitials. Temper rolling 2% increased the 0.2% proof stress and the bake hardening treatment had no effect on the yield behaviour, again indicating the removal of interstitials during the batch annealing treatment.

Table IV shows a comparison of the ratios of the intensities of the (222) component to the (200) component and the average anisotropy ratios of 70% cold rolled and heat treated steels. The steels show little texture, which is in good agreement with the low $r_m$ values as shown by Held [14].

## 5. DISCUSSION

Usually the yield point can be associated with small amounts of interstitial or substitutional impurities. Only 0.0001% of carbon or nitrogen is required for the appearance of a yield point [8]. It is possible that the cooling rate after hot rolling was not sufficiently slow to allow full precipitation of all carbides, (TiC, $V_4C_3$ or NbC), and allowed a marginal residual interstitial content, thus giving the observed variation in the yield behaviour in steels 2-7.

For the cold rolled and annealed steels, steels 2,3,5 and 6 containing vanadium all showed discontinuous yielding and steels 4 and 7 containing titanium or titanium and niobium showed continuous yielding.

It has been shown [9] that if carbon and nitrogen are removed from steel, both the initial discontinuous yielding and strain ageing are eliminated. The pinning of dislocations is an important feature of the yield point phenomenon. Pinning can arise from the solute-dislocation interaction or by precipitation of fine carbides or nitrides along the dislocation. The yield point may occur as a result of unlocking the dislocations by a high stress, or in the case of strong pinning, by creating new dislocations at points of stress concentration.

Dislocation locking by interstitial atoms is an important aspect of yield point behaviour and even though much of the carbon and nitrogen may have been fixed by the carbo-nitride forming elements, discontinuous yielding behaviour may still be seen if only small amounts of interstitials remain [10].

Temper rolling removes the yield point by providing new dislocations without atmospheres. Strain ageing and bake hardening only occurs where there are interstitial elements in solution. Nitrogen produces a more noticeable strain ageing effect than carbon. The rate of strain ageing, the final yield stress and the tensile strength increase with increasing interstitial content. The effectiveness of carbide and nitride formers in preventing strain ageing increases in order of their affinity for carbon and nitrogen ie. V, Nb, and Ti. There is a greater affinity for nitrogen as the nitrides tend to be more stable [11], although aluminium was added to all the steels, and aluminium nitride would be

**Table IV : Ratio of intensity of (222) to (200) texture component and $r_m$ values of 70% cold rolled and heat treated steels 1-7.**

| Steel No. | $I_{(222)}/I_{(200)}$ | $r_m$ |
|---|---|---|
| 1 | 1.25 | 0.82 |
| 2 | 0.95 | 1.00 |
| 3 | 1.25 | 0.69 |
| 4 | 1.20 | 1.06 |
| 5 | 0.83 | 0.86 |
| 6 | 1.00 | 0.63 |
| 7 | 1.30 | 0.91 |

Table V : Tensile test data for hot band (batch) annealed and 80% cold rolled heat treated steels 1-7.

| Process | Steel No. | Elong/% | LYP/MPa | 0.2%PS/MPa | UTS/MPa | YP El/% | $r_m$ |
|---|---|---|---|---|---|---|---|
| Hot Band (Batch) Annealed | 1 | 50 | - | 88 | 275 | - | - |
| | 2 | 52 | - | 127 | 284 | - | - |
| | 3 | 51 | - | 113 | 279 | - | - |
| | 4 | 48 | - | 103 | 275 | - | - |
| | 5 | 52 | - | 104 | 269 | - | - |
| | 6 | 52 | - | 102 | 271 | - | - |
| | 7 | 50 | - | 115 | 283 | - | - |
| 80% Cold Rolled & Heat Treated 800°C for 30s | 1 | 43 | 219 | - | 327 | 6.2 | 1.46 |
| | 2 | 37 | 275 | - | 343 | 7.0 | 1.70 |
| | 3 | 43 | 248 | - | 325 | 7.6 | 1.35 |
| | 4 | 40 | 125 | - | 326 | 0.5 | 2.23 |
| | 5 | 44 | 259 | - | 325 | 7.7 | 1.47 |
| | 6 | 41 | 261 | - | 335 | 7.6 | 1.37 |
| | 7 | 39 | 138 | - | 366 | 0.5 | 1.76 |
| 80% Cold Rolled 644°C for 60s | 1 | 39 | 175 | - | 358 | 2.4 | 1.36 |
| 80% Cold Rolled 656°C for 60s | 2 | 39 | 294 | - | 374 | 7.7 | 1.49 |
| 80% Cold Rolled 673°C for 60s | 3 | 44 | 201 | - | 353 | 3.4 | 1.56 |
| 80% Cold Rolled 697°C for 60s | 4 | 41 | - | 128 | 349 | - | 1.85 |
| 80% Cold Rolled 707°C for 60s | 5 | 42 | 155 | - | 353 | 0.5 | 1.53 |
| 80% Cold Rolled 711°C for 60s | 6 | 42 | 199 | - | 355 | 3.1 | 1.28 |
| 80% Cold Rolled 755°C for 60s | 7 | 43 | 129 | - | 367 | - | 1.42 |

the most stable nitride in the vanadium steels.

The average anisotropy ratio, $r_m$, depends on the orientation texture present within sheet steel. Held [14] has shown that high r values are produced by textures containing a high proportion of the (111) texture component and a low proportion of the (100) texture component. Interstitial carbon and nitrogen in solution during cold rolling can have an important effect on structural development [15]. A steel with high interstitial carbon content can dynamically strain age during rolling, leading to shear band formation in grains of (111)[112] orientation and lead to easy nucleation during annealing to promote the formation of a (110) orientation at the expense of the (111) orientation.

The influence of dissolved carbon and nitrogen during annealing is mainly to increase the proportion of minor components and decrease the proportion of (111) components which contribute to good drawability [16]. Higher interstitial contents correlate with poor texture, a lower $r_m$ value and a finer grain size [17]. On cooling after annealing, interstitials taken into solution during the early stages of annealing will be reprecipitated on undissolved carbo-nitrides. If the number of carbo-nitrides is small and their spacing large or all the carbo-nitrides have been taken into solution precipitation becomes more difficult and residual carbon remains in solution. The precipitation of carbon is more difficult in continuously annealed steels due to the short times involved. For interstitial free steels it is the absence of interstitial carbon and nitrogen that contributes to the favourable textures seen in these steels [17 & 18].

In order to check that the interstitial free condition of all the microalloyed steels would in fact develop suitable cold rolled and annealed textures further experimental work was carried out on material that had been hot band (batch) annealed prior to cold rolling. The results for the annealed hot band are shown in Table V. These show quite clearly that the interstitials have been successfully scavenged by the microalloys, and all steels showed continuous yielding behaviour. Even the plain carbon steel showed continuous yielding behaviour as the carbon would precipitate as cementite, and the nitrogen would form aluminium nitride. These steels were then cold rolled to give an 80% reduction in thickness to enhance the texture development, and continuously annealed at a temperature just above the recrystallisation temperature. Other cold rolled material from all steels was continuously annealed at a standard temperature of 800°C. The tensile properties of these 80% cold rolled and annealed steels (following hot band annealing) are also shown in Table V. The mechanical properties were virtually identical with those observed previously, with the exception of the value of $r_m$. In all cases the plastic anisotropy index had increased and in steel 4 (the titanium steel) had attained a value of 2.2 after continuous annealing at 800°C. The highest levels of plastic anisotropy were seen in the titanium and titanium-niobium steels, but reasonably high values of $r_m$ were attained in the vanadium steels, those being in the range 1.3-1.7. Thus, it has been demonstrated that the high levels of plastic anisotropy are attained by cold rolling in the interstitial free condition.

It is interesting to note that continuous yielding is only seen in the titanium steel, but low yield points with small luders extensions were also seen in the titanium-niobium and one of the titanium-vanadium steels (steel 5). It would be expected that significant bake hardening would be attainable in the vanadium and vanadium-titanium steels after the final continuous annealing treatment, this being accompanied by good levels of plastic anisotropy.

## 6. CONCLUSION

For hot rolled steels discontinuous yielding predominated, though for steels 4,5 and 7 (containing Ti) some continuous yielding behaviour was seen, indicating the scarcity of interstitials in solution after hot rolling. For the simulated continuous annealed steels, steels 1,2,3,5 and 6 showed discontinuous yielding behaviour (V and V-Ti alloys) indicating the presence of interstitials retained in solution, whilst steels 4 and 7 showed continuous yielding behaviour (Ti and Ti-Nb alloys) indicating the removal of interstitials from solution. Samples showing discontinuous yielding had higher yield points compared to those showing continuous yielding behaviour, but comparable ultimate tensile strengths. Recrystallisation of the cold worked material lead to a reduction of hardness by approximately 100 $Hv_{10}$ and a microstructure with a smaller grain size than the initial hot rolled material thus giving higher yield and ultimate tensile strengths. The simulated continuous annealed steels showed little texture formation and consequently low plastic anisotropy values.

Vanadium and vanadium-titanium steels recrystallised at temperatures below those of the titanium-niobium steels. Titanium is a more efficient carbo-nitride forming element than vanadium, and it is thought that during the thermal processing of these steels the vanadium did not have sufficient time to combine with the carbon and nitrogen, or was present in insufficient quantity, to form vanadium carbo-nitrides.

Batch annealing removed all the interstitials from the hot rolled steels which all showed continuous yielding behaviour. The removal of carbon and nitrogen interstitials is thought to be by carbo-nitride and/or cementite formation during the slow cooling.

The processing variables for interstitial steels have been shown to be critical. Vanadium additions permit lower annealing temperatures for full recrystallisation of cold rolled strip. Although the cold rolled and annealed strip showed a discontinuous yield point a 2% temper roll produced continuous yielding characteristics and resulted in extended periods before the return of the yield point phenomenon with good bake hardening in vanadium steels.

The hot rolling variables and cold rolling reductions (70%) were not optimal for these steels giving initially disappointingly low values of plastic anisotropy. It has been shown that the removal of interstitial elements from solution in the hot band prior to cold rolling resulted in reasonable values of plastic anisotropy. The vanadium steels showed reasonable values of plastic anisotropy together with good bake hardening potential.

Further work is in hand to examine the effects of low soaking temperature in combination with slow cooling of coiled hot band to remove interstitials from solution.

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