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ACHIEVING GRAIN REFINEMENT THROUGH RECRYSTALLIZATION CONTROLLED ROLLING AND CONTROLLED COOLING IN V-Ti-N MICROALLOYED STEELS

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ABSTRACT

A new thermomechanical processing route, labeled recrystallization controlled rolling (RCR), and the alloy design philosophy corresponding to this processing are proposed. RCR avoids the high mill loads and low productivity associated with conventional controlled rolling (CCR) and is based upon (i) achieving a very fine as-reheated austenite, (ii) repeated deformation and recrystallization above the recrystallization temperature of austenite and (iii) accelerated cooling to some intermediate temperature, followed by air-cooling to room temperature. Steel compositions which are ideal for this processing should have a relatively high grain coarsening temperature during reheating, T_{GC}, a relatively low recrystallization temperature during rolling, T_{RX}, and a low grain coarsening rate after deformation, R_{GC}. In addition, the steels should exhibit an adequate capacity for undercooling and precipitation hardening of the ferrite.

Experiments conducted on .13V and .13V-.017Ti steels have shown that a very small addition of Ti to V steels markedly raises the T_{GC}. The T_{GC} of the V-Ti steels in the as-slabbed condition increases with increasing N content while the T_{GC} of the same steel in the as-cast condition is insensitive to N content. Nevertheless, in all cases, the former is much lower than the latter. In both the .13V and .13V-.017Ti steels, the lowest temperature at which austenite completely recrystallizes in a short time after a 50% reduction, the T_{RX}, increases with increasing N level; however, the T_{RX} of the V-Ti steels is much lower than that of the V steels if the processing parameters are the same. The temperature difference (T_{GC}-T_{RX}), the temperature range of RCR, is at least 175°C higher in the V-Ti steels than in the V steels. In the .13V-.017Ti-.012N steel, an effective austenite interfacial area, S_V, of approximately 160 mm^{-1} can be generated by rolling in the range of 850 to 1080°C. This S_V is remarkably temperature insensitive and resulted from recrystallized austenite in all cases. The hot strength in the second hit of interrupted compression tests at 850°C and a strain rate of 5 s^{-1}, to which mill loads are directly related, is 60% higher in the .13V-.025N steel than in the .13V-.017Ti steels. In addition, the V-Ti steels show strong inhibition of grain growth after hot rolling due to the pre-existing fine TiN particles. Uniform and fine ferrite grain sizes to 6 microns can be achieved using RCR. The precipitation potential in α of V and V-Ti steels increases with increasing N content as well as cooling rate. An addition of .017 wt% Ti to V steels decreases the precipitation potential; however, the loss can be reduced by using a relatively high N level and accelerated cooling.

While extensive grain refinement is achievable using CCR, the high mill loads and low productivity have kept this process from being widely applied in North America as well as in most of the rest of the world. The attractiveness of RCR is that it is a rolling procedure which can result in extensive grain refinement, and, which at the same time, can be immediately employed on existing rolling facilities.

IT IS WELL KNOWN that a fine ferrite grain size in the final product can be achieved by proper hot rolling. Recent work has focused on the ferrite grain refinement which results from the conventional controlled rolling (CCR) of austenite. As much of the deformation in CCR takes place below the recrystallization temperature, T_{RX}, high ferrite nucleation rates are promoted. This is due to the high effective interfacial area, S_V, which results principally from the change in austenite grain shape and from the generation of deformation bands.[1] Unfortunately, this rolling is done at low temperatures and is associated with high mill loads.[2-5] In addition, the productivity of CCR is low because of the lengthy delay times between the roughing and finishing passes required to reach the low finish rolling temperature.[3] The present work attempts to develop a high S_V in the austenite and hence a fine ferrite grain size without the high mill loads and low productivity associated with CCR.

The proposed alternative to CCR is based upon (i) achieving a very fine as-reheated austenite grain size (ii) repeated deformation and recrystallization above the T_{RX}, and (iii) accelerated cooling to some intermediate temperature, followed by air cooling to room temperature. This new process is labelled recrystallization controlled rolling (RCR).

Steel compositions which would be ideal for this proposed processing should have certain metallurgical characteristics, i.e. a relatively high grain coarsening temperature, T_{GC} a relatively low recrystallization temperature during rolling, T_{RX}, and a low grain coarsening rate after rolling, R_{GC}, as well as an adequate capacity for under cooling and precipitation hardening of the ferrite.

It is well known that the T_{GC} can be raised and the R_{GC} can be lowered by the presence of fine, stable particles.[6,7,8] In fact, the stability of the precipitate determines both the T_{GC} and the temperature range over which the R_{GC} is a minimum. It is also well known that the recrystallization temperature after hot rolling is directly related to the amount and size of the strain-induced precipitate.[9-14] That is, high recrystallization temperatures are promoted by large amounts of fine strain induced precipitates. Conversely, steels with low recrystallization temperatures, similar to those of plain carbon steels, should not have a large capacity for strain induced precipitation. The V-Ti-N system appears to be ideally suited for this purpose, since TiN will precipitate at very high temperatures,[15-20] thereby reducing the N level of the austenite and inhibiting VN precipitation, and VC is not known to precipitate during low temperature rolling. Hence, the V-Ti-N system should exhibit a high T_{GC}, a low R_{GC}, and a low T_{RX}. Finally, the vanadium left in solution in the austenite would be available to form precipitates during the ferrite transformation, imparting greater strength to the final product.

EXPERIMENTAL PROCEDURE

The steels used in this investigation were produced as 22 kg laboratory heats. The V-Ti steels were aluminum killed and the V steels were silicon killed. Chemical compositions are given in detail in Table 1.

The ingots were reheated and hot rolled to plates 40mm thick and to bars 16mm in diameter. Specimens for reheating (cubes 10mm on a side) and hot rolling experiments (blocks 50mm x 50mm and 16mm thick) were machined from the plates. Specimens for hot compression experiments (cylinders 12mm in diameter and 18mm in height) were machined from the rounds. Therefore, all hot deformation studies were conducted on slabbed materials.

Reheating was performed under an argon atmosphere in a box furnace. Materials were available in both the slabbed and as-cast condition.

Rolling experiments were carried out on a laboratory mill operating at a strain rate of 4.9 or 6.9 s^{-1}. After rolling, the specimens were water quenched, air-cooled, or subject to accelerated cooling by forced air or water spray. In this fashion, cooling rates of 2, 7, and 15°C s^{-1} could be obtained as desired.

Hot compression experiments were carried out on an MTS machine modified for constant true strain rate testing.[21] Barreling was avoided by using a modified Rastegaev's grooving technique[22] and applying glass lubricant (Deltaglaze 347 or 349) to both specimen and dies. Most hot compression experiments were performed at constant true strain rates of 2 and 5s^{-1}. The specimens could be quenched within a fraction of a second for metallographic observation. In some cases, interrupted compression tests were

Table 1 Chemical Composition of Experimental Steels, wt%

Steel	C	Mn	Si	Al Total	Al Soluble	N	V	Ti
V	.08	1.25	.43	.002	-	.006	.13	-
V-N	.08	1.23	.43	.002	-	.025	.13	-
V-Ti	.07	1.24	.42	.034	.017	.006	.13	.017
V-Ti-N	.08	1.22	.40	.032	.014	.012	.13	.016

$P \leq 0.05$; $S \leq 0.05$ for all steels carried out in which the specimen is given a certain amount of strain at a prescribed temperature and strain rate, ε_{PRE}. The deformed specimens are then unloaded and held at the deformation temperature for various times to allow for microstructural change. The sample is then reloaded and deformed further at the temperature and strain rate of the pre-strain. In order to assess both the yield stress and rate of work hardening, an average flow stress,

$$\bar{\sigma} = \frac{1}{0.3} \int_0^{0.3} \sigma(\varepsilon)\, d\varepsilon$$

was used as a measure of hot strength in both the first and second hits.

When applicable, the specimens were examined by optical metallographic techniques. A linear intercept method was used to determine the grain size. A modified picric acid was used to reveal the prior austenite grain boundaries and nital was used to reveal the ferritic microstructures.

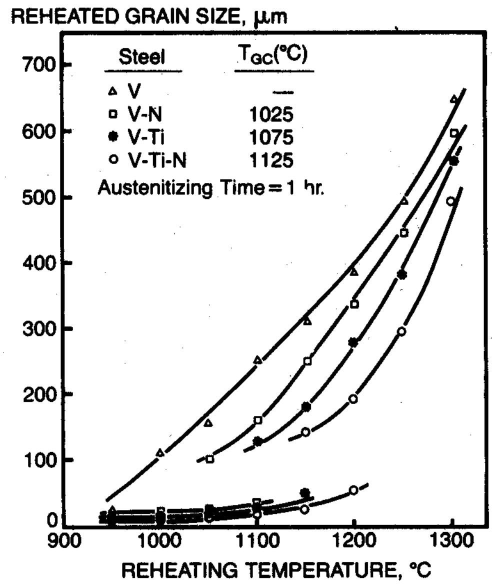

Figure 1 Grain coarsening behavior of V and V-Ti steels during reheating.

RESULTS AND DISCUSSION

REHEATING BEHAVIOR-Results of the reheating studies are given in Figure 1. The as-reheated grain size for the four steels is shown as a function of temperature for a common heating time of one hour. The V steel exhibits normal grain growth over the entire temperature range. On the other hand, the V-N, V-Ti, and V-Ti-N steels show three stages of grain coarsening, as previously reported.(7) In addition, the onset of abnormal grain growth is conservatively estimated and tabulated as the grain coarsening temperature, T_{GC}, in Figure 1. The effect of N level on T_{GC} is shown by the uppermost curves in Figure 2.

Concerning the grain coarsening temperature, there are three phenomena of note. Firstly, a very small (.017 wt.%) addition of Ti to V steels raises the T_{GC} markedly, Figures 1 and 2. This is caused by the presence of fine and stable TiN particles in the V-Ti steels.

Secondly, the T_{GC} of the V-Ti steels is much lower in the as-slabbed condition than in the as-cast condition. As hot rolling marginally changes the size distribution of TiN particles, this lowering of T_{GC} was explained as a consequence of a direct relationship between mean grain size and critical mean particle size in Ti alloyed steels.(15-16) In V-Ti steels, it was explained as a result of the dissolution of fine V(C,N) particles.(23) Current experiments conducted on a V-Ti steel in the as-cast condition show that after reheating to 1100°C, a single 60% reduction at 1050°C, followed by immediate reheating again also reduces the T_{GC}.(24) In this case and the case of V-free Ti steels, there were no VN particles involved in boundary pinning and ripening. Also, the difference between the average austenite grain size of as-cast and as-slabbed materials (2-4 microns) would not be expected to cause such a great difference (>100°C) in the T_{GC}. It is clear that further investigation is needed to show how hot rolling and γ/α transformation compromise the role of the TiN particles or change the mobility of austenite grain boundaries.

Thirdly, an increase in the N level in the range investigated leads to an increase in the T_{GC} of the V-Ti steels in the slabbed condition but does not affect the T_{GC} of the same steel in the as-cast condition. In other words, a nitrogen content greater than the stoichiometric one for TiN, which is about .005 wt.% in the case of .017 wt.% Ti, is beneficial or at least not deleterious to the T_{GC} of V-Ti steels. The different influence of N level on T_{GC} of the V-Ti steels in the as-cast and as-slabbed conditions may be described as follows. At temperatures close to the T_{GC} of the as-cast materials, the VN particles have already dissolved in the austenite. From this, the T_{GC} of the as-cast materials depends primarily on the volume fraction of fine TiN particles. In the range investigated, when the N level is increased, the volume fraction of total TiN particles will increase also, but the volume fraction of the fine TiN particles precipitated after solidification may change only slightly. One possible explanation for this behavior is because the dissolution temperature of the TiN in the V-Ti steel is not far from the solidification stop temperature. Hence, an increase in total nitrogen will be taken up mainly by the large TiN which would precipitate in the liquid during solidification. Therefore, the T_{GC} of the V-Ti steels in the as-cast materials is relatively insensitive to the N content of the steels. On the other hand, at temperatures close to the T_{GC} of the slabbed materials, which

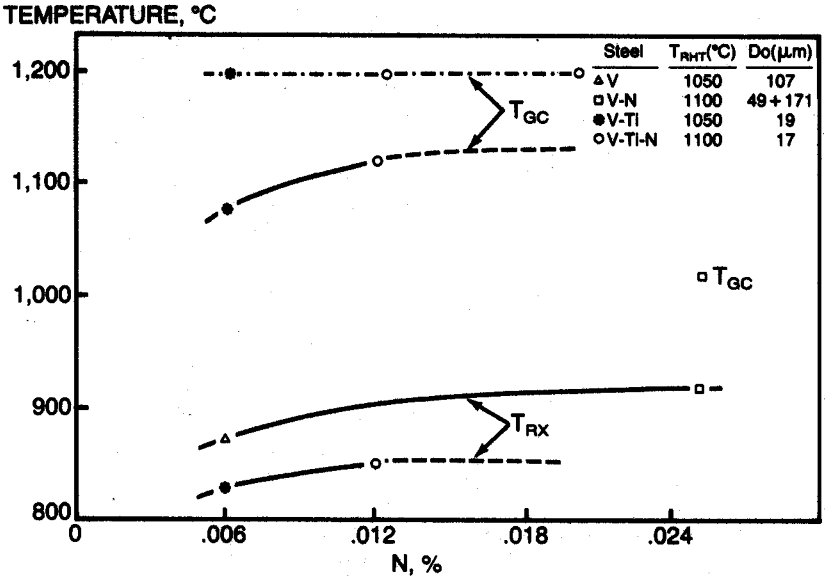

Figure 2 Effect of N on T_{GC} AND T_{RX}. The upper T_{GC} curve refers to as-cast material, all others refer to slabbed material.

is lowered by hot deformation and austenite/ferrite transformation, a large number of VN particles still exist. In this case, the T_{GC} is related to boundary pinning by the VN particles. The higher the N level of the steels, the greater the volume fraction of total VN particles, and hence the higher the T_{GC} of the steels in the slabbed condition.

RESPONSE TO HOT DEFORMATION-The experimental procedures used to determine the T_{RX}, d_γ and S_V of interest were as follows. Specimens 16mm thick were reheated for 30 minutes to a variable temperature, T_{RHT}, allowed to cool at a rate of $1°C\ s^{-1}$ to the deformation temperature, T_{DEF}, reduced 50% by rolling upon reaching T_{DEF}, and thereafter quenched by immersion in agitated iced brine. Transfer of specimens from the rolling table to the quenching vessel was achieved within 3 seconds. The state of the austenite after 50% reduction by rolling was determined by optical microscopy of quenched specimens. The results are summarized for the four steels in Table 2 as a function of T_{DEF}. From these data, the lowest temperature at which austenite completely recrystallizes after a single 50% reduction, the T_{RX}, was obtained, and is given by the bottom curves of Figure 2. Furthermore, the effect of temperature and strain rate on the as-rolled austenite grain size and the effective austenite interfacial area, S_V, are presented in Figure 3.

Table 2 Recrystallization State

Steel	T_{RHT}, °C	D_o, μm	Rolling Temp., °C									
			1080	1030	982	954	927	899	871	850	830	788
V-Ti	1050	19.2		R	R	R	R	R	R	R	P	N
V-Ti-N	1100	17.0	R		R	R	R	R	R	P	P	N
V	1050	107		R		R		R	P	P	N	N
V-N	1100	49 + 171			R	R	P	P	N	N	N	N

R = recrystallized P = partly recrystallized N = non-recrystallized

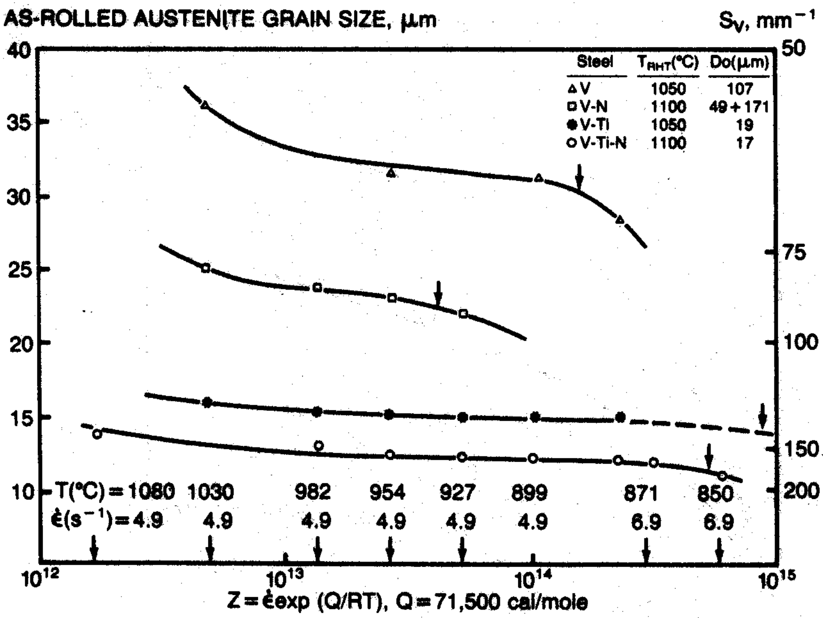

Figure 3 Influence of rolling condition at 50% red. on the as-rolled asutenite grain size and interfacial area S_V. The T_{GC} is marked on each curve.

In both the V and V-Ti steels, the T_{RX} increases with increasing N level (Figure 2) although the initial grain size is smaller in the high N steels than in the low N steels. Apparently, this is caused by increasing the amount of supersaturation of N in austenite, enhancing the strain-induced precipitation of VN in austenite for the V steels,[12,13,14] and V-Ti-N steel.[24] However, under the condition of constant total N level, the T_{RX} is significantly lower in the V-Ti steels than in the V steels if the process parameters are the same. Thus, the temperature difference (T_{GC}-T_{RX}), i.e. the temperature range for RCR, is at least 175°C greater in the V-Ti steels than in the V steels. The cause of low T_{RX} in the V-Ti steels is attributed to (i) the decrease in N content of austenite due to the previous precipitation of TiN hence reducing the strain-induced precipitation of VN in austenite, and (ii) the fine initial austenite grains obtained by reheating below the T_{GC}. So far, no evidence has been obtained to show that the pre-existing TiN particles can initiate austenite recrystallization.

For both the V and V-Ti steels, the as-rolled grain size decreases and the S_V increases with increasing N level, Figure 3. However, a much smaller as-rolled grain size and a much greater S_V can be generated by a single 50% reduction in the V-Ti steels than in the V steels. For instance, a grain size of 12.5 microns and an S_V of 160 mm^{-1} were achieved by rolling above the T_{RX} of the V-Ti-N steel. This grain size and S_V are remarkably temperature insensitive in the range of 850 to 1080°C, and resulted from recrystallized austenite in all cases. The temperature insensitivity of S_V for the V-Ti-N system allows for the possibility of obtaining more uniform structure and properties in plate products. The behavior of the V steels is quite different. The S_V is much lower and is temperature sensitive, Figure 3.

Recent hot compression test results[24] show that a 50% reduction at a mean strain rate of 4.9 s^{-1} exceeds the critical strain for the onset of dynamic recrystallization at 860°C and reaches the critical strain for completion of dynamic recrystallization at 1020°C. This indicates that dynamic recrystallization, under the present experimental conditions, plays a more important role in RCR of V-Ti-N steels than in the conventional rolling or CCR of V or Nb steels where the initial grain size is great and hence the two critical strains are high.[25]

Measurements of the grain growth rate of austenite following deformation were performed on the four steels. All specimens were reduced 55% (ε=0.8) in uniaxial compression at a constant deformation temperature of 1050°C, and at a constant true strain rate of 2 s^{-1}. Under these conditions all the materials were in the steady state regime of flow. Following straining, the specimens were held at 1050°C for various times before water quenching. No abnormal grain growth was observed. The measured grain size as a function of holding time is plotted in Figure 4. The rates of grain growth are obtained from the curves and presented in Table 3.

A small addition of Ti to the V steels results in a drastic decrease in the R_{GC}. Significantly, the rate of the Ti-bearing steels is negligible beyond 20 seconds holding at 1050°C.

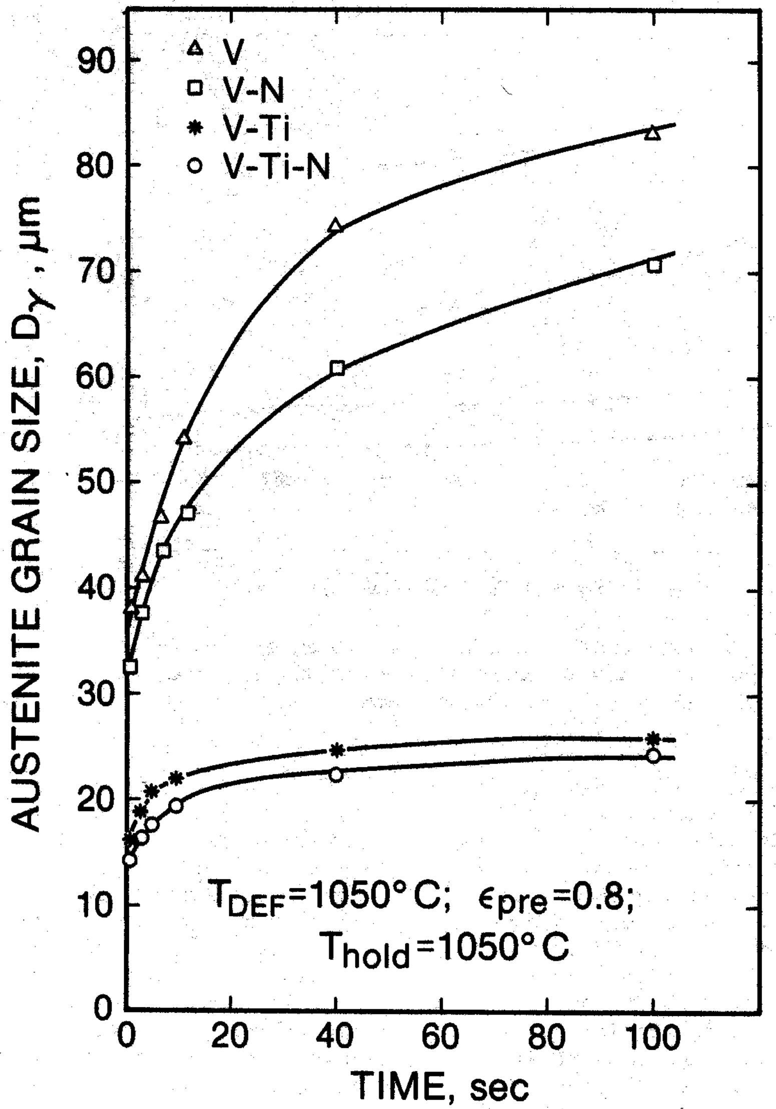

Figure 4 Grain coarsening behavior after hot deformation and recrystallization.

Table 3 Grain Coarsening Rate

Steel	Average R_{GC} after Recrystallization, μm/sec		
	in 1st 20 sec	in 2nd 20 sec	after 40 sec
V	1.30	0.58	0.17
V-N	1.08	0.42	0.17
V-Ti	0.38	0.075	0.016
V-Ti-N	0.37	0.065	0.016

Analogous results were reported by C. Ouchi, et al.[8] for Ti steels and W. Roberts, et al.[26] for V-Ti steels. Another phenomenon of note is that the R_{GC} of the V-Ti steels is not sensitive to N level in the range investigated. The reason for this may be the same as suggested in the discussion of N level on T_{GC} of the V-Ti steels.

The average flow stresses of the four steels were measured by means of interrupted compression tests at 850°C and strain rate of 5 s^{-1} with a pre-strain of 0.5. A great difference in the hot strength of recrystallized and non-recrystallized austenite can be revealed using this technique. The results are given in Table 4. In the first hit of the compression tests, the average flow stresses for the four steels are almost the same (V steel has slightly lower hot strength). In the second hit, however, they are quite different. After 3 seconds holding, the stress of the V-N steel in the second hit is 24% higher than that of the V-Ti steel. After 6 seconds holding, the stress of the V-N steel is 60% higher than that of V-Ti steel. There is no softening at all in the V-N steel during holding.[24] The volume fraction of recrystallized austenite corresponding to these short times is zero in the V-N steel, i.e. 100% pancaked, and is respectively about 70% and 80% in the V-Ti steel. Thus, the high average flow stresses observed in the second hit of the V-N steel are caused by the pancaking of austenite and by precipitation hardening in austenite. It follows that the high mill loads associated with CCR are directly related to similar high average stresses. However, if the V-Ti steels are reheated below the T_{GC}, the austenite is able to recrystallize in a short time even at 850°C and does not have a strong precipitation potential. The result is that the average flow stress on the second hit is low, and hence the mill load in RCR of the V-Ti-N system should be low too, even at 850°C.

GRAIN REFINEMENT THROUGH GAMMA/ALPHA TRANSFORMATION-The grain refinement ratio, d_γ/d_α, of the four steels was investigated as follows. Specimens were reheated to the temperatures shown in Figure 5, reduced 50% by rolling at 900°C, cooled at rates of 2, 7, and 15°C s^{-1} to 560°C, and air-cooled to room temperature. The d_α was measured from the specimens. The d_γ was obtained from the data shown in Figure 3. The d_γ/d_α as a function of d_γ and cooling rate is plotted in Figure 5. The effect of N content on ferrite grain size is presented in Figure 6. A comparison of the ferrite grain size distribution of the RCR V-Ti-N steel and a CCR Nb steel is provided in Figure 7. Included in these data are the average grain diameters and the standard deviations.

The ratio, d_γ/d_α, decreases with decreasing austenite grain size and cooling rate. When the d_γ is less than 10 microns (or S_V>200 mm^{-1}) and the cooling rate is lower than 2°C s^{-1}, the ratio is close to one, i.e. there is almost no grain refinement through the γ to α transformation in these steels. The transformed ferrite grain size is also related to N content, Figure 6. Uniform and fine ferrite grain size, down to 6.3 microns, can be achieved using the rolling schedule described. This average ferrite grain diameter is 1 micron greater than that of typical CCR material; however the standard deviation of the diameter of the former is 1 micron less than that of the latter, Figure 7. Some especially large grains (diameters up to 18 microns) exist in the CCR material. This non-uniformity has a deleterious effect on toughness.[27] The RCR of V-Ti-N steels will apparently permit this unacceptable microstructural feature to be avoided.

PRECIPITATION HARDENING IN FERRITE-The TMT used to investigate the precipitation potential of the four steels is the same as that used to study the d_γ/d_α ratio. The intensity of the precipitation strengthening in the steels was evaluated by the difference in the hardness of microalloyed steel and a C-Mn steel of the same ferrite grain size.[28] The results are shown in Figure 8 as a function of N content and cooling rate. The precipitation hardening increment increases with increasing N level and cooling rate between 900 and 560°C in all the

Table 4 Average Flow Stress

Steel	Average Flow Stress* (MPA) at 850°C and 5 sec^{-1} in 1st Hit	in 2nd Hit after Holding for 3/6 sec
V-Ti	146	163/157
V-Ti-N	144	166/161
V	130	176/162
V-N	146	201/250

*Average Flow Stress = $\int_0^{0.3} \sigma(\varepsilon)d\varepsilon/0.3$

steels. An addition of .017 wt% Ti to the V steels decreases the precipitation potential; however, the loss can be reduced by using a relatively high N content and interrupted accelerated or controlled cooling. Similar results have been reported for V steels[29] and V-Ti steels.[23]

Recent hot compression test results show that the onset of the VN precipitation occurs at least an order of magnitude earlier in the V-Ti-N steel than in the V steel.[24] This is attributed to the pre-existing TiN particles which may initiate the precipitation of VN in austenite and has been observed by T. Siwecki, et al.[23]

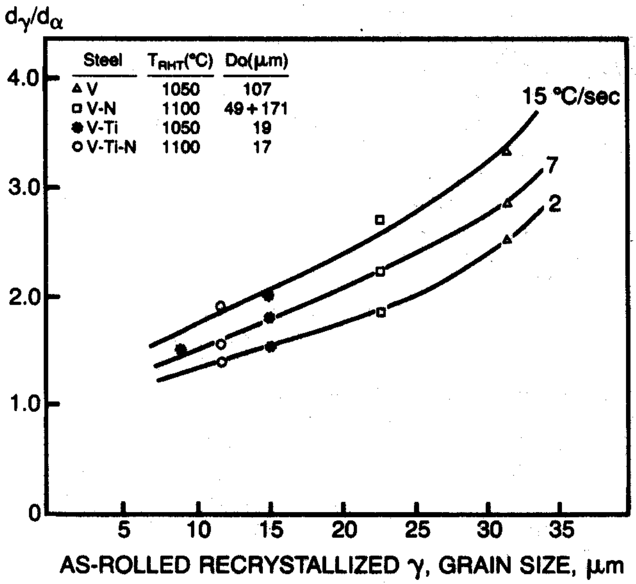

Figure 5 Dependence of grain refinement ratio on austenite grain size and cooling rate.

CONCLUSIONS

RCR of microalloyed steels and the alloy design philosophy corresponding to this processing have been proposed and discussed. The reheating behavior, response to hot deformation, grain refinement through the γ to α transformation and precipitation hardening in α of two .13V-.018Ti steels and two reference .13V microalloyed steels have been investigated. The results are summarized as follows.

1.) The proposed RCR is based upon (i) achieving a very fine as-reheated austenite grain size, (ii) repeated deformation and recrystallization above the T_{RX}, and (iii) accelerated cooling to some intermediate temperature followed by air-cooling to room temperature. Steel compositions which are ideal for this RCR processing should have certain, specific metallurgical characteristics; i.e. a relatively high T_{GC}, a relatively low T_{RX} during rolling, and a low R_{GC} after deformation. In addition, the steels should exhibit an adequate capacity for undercooling and precipitation hardening of the ferrite.

2.) A very small addition of Ti (.017 wt%) to the V steels raises the T_{GC} markedly. The T_{GC} of the V-Ti steels in the as-slabbed condition increases with increasing N content while the T_{GC} of the same steels in the as-cast condition is insensitive to N content. Nevertheless, in all cases, the former is much lower than the latter.

3.) In both V and V-Ti steels the lowest temperature at which austenite completely recrystallizes in a short time after 50% reduction, the T_{RX}, increases with the increasing N level; however, the T_{RX} of the V-Ti steels is much lower than that of the V steels if the process parameters are the same. Thus the temperature difference ($T_{GC}-T_{RX}$), i.e. the temperature range for RCR, is at least 175°C greater in the V-Ti steels than in the V steels.

4.) In the .13V-.017Ti-.012N steel, an

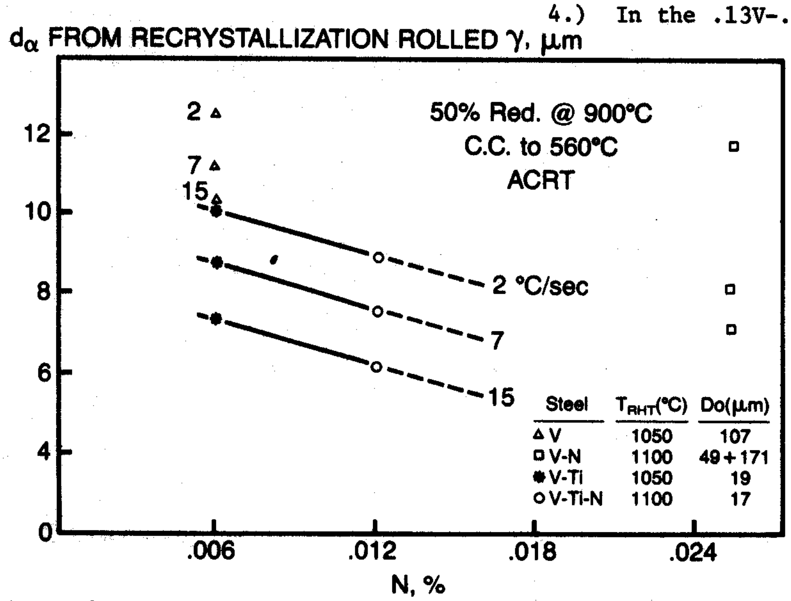

Figure 6 Dependence of ferrite grain size on N and cooling rate.

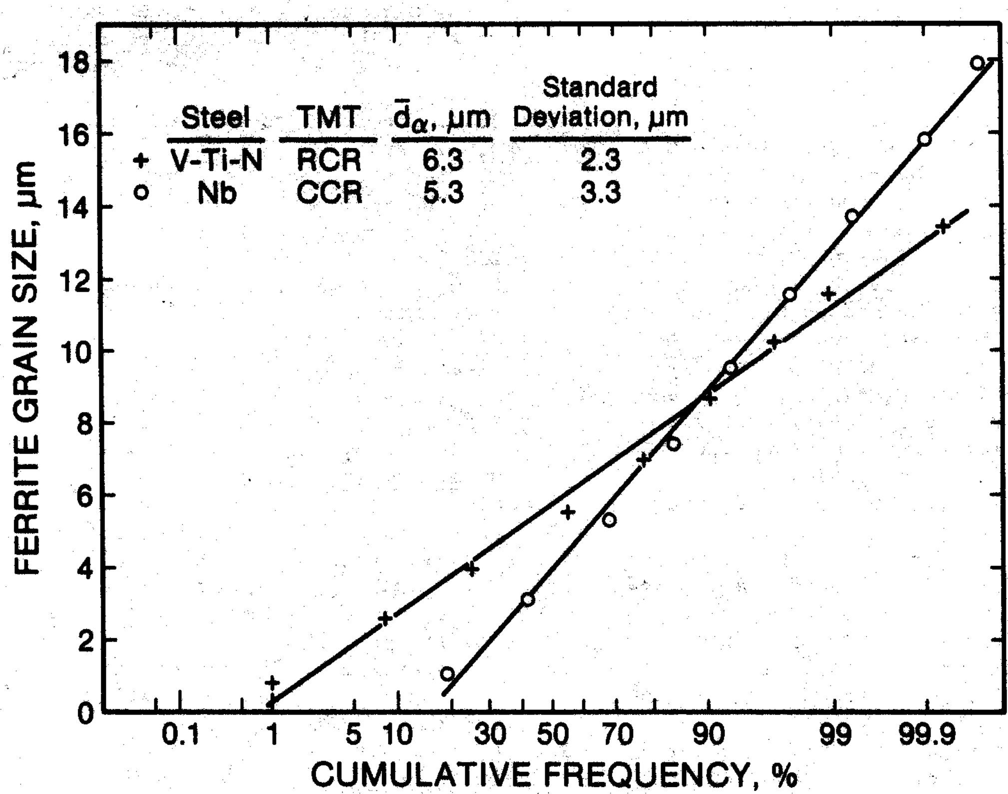

Figure 7 Ferrite grain size distribution of RCR V-Ti N steel and CCR Nb steel.

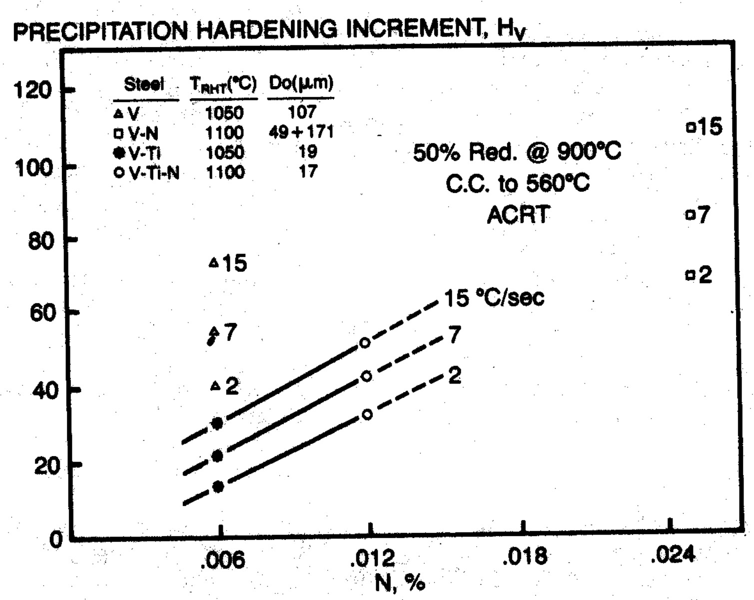

Figure 8 Dependence of precipitation hardening increment on N and cooling rate.

effective interfacial area, S_v, of approximately $160 mm^{-1}$ can be generated by rolling in the range of 850 to 1080°C. This S_v is remarkably temperature insensitive in all cases.

5.) The V-Ti steels show strong inhibition of grain growth after hot rolling due to the preexisting fine TiN particles.

6.) The average flow stresses at 850°C and a strain rate of $5\ s^{-1}$ in the first hit are almost the same for the V steels and the V-Ti steels; however, in the second hit the stress is 60% higher in the pancaked .13V-.025N austenite than in the recrystallized .13V-.017Ti austenite. The low average stress in the second hit for the V-Ti steels is associated with both the ability of the fine austenite to recrystallize rapidly and the weak strain-induced precipitation potential. The mill loads, directly related to the hot strength of materials, would be expected to be low during the RCR of V-Ti steels.

7.) The grain refinement ratio, d_γ/d_α, depends on the austenite grain size prior to transformation and cooling rate following rolling. When d_γ is less than 10 microns (S_v>200 mm^{-1}) and the cooling rate is lower than $2°C\ s^{-1}$, the ratio is close to one, i.e. there is almost no grain refinement through the γ to α transformation. Uniform and fine ferrite grain sizes to 6 microns can be achieved using RCR.

8.) The precipitation potential during and after the γ to α transformation of the V and V-Ti steels increases with increasing N content as well as cooling rate. An addition of .017 wt% Ti to V steels decreases the precipitation potential; however, the loss can be reduced by using a relatively high N level and accelerated or controlled cooling.

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