

FERRITE GRAIN REFINEMENT IN SEAMLESS PIPES THROUGH INTRAGRANULAR NUCLEATION ON VN

S. Zajac, B. Hutchinson, R. Lagneborg, M. Korchynsky

Mill rolling process of seamless pipe was simulated with the aim of applying grain refinement through the formation of intragranular ferrite on VN precipitated inside austenitic grains, for production of as-hot rolled microstructures, which are currently attained only by in-line normalising. Tests were performed on V+N and V+N(Ti) steels using two types of schedules with long and short transfer/heating times prior to sizing between 930-830°C. It was found that transfer/heating time between high temperature rolling and low temperature sizing could be used for precipitation of VN in austenite which in turn can nucleate intragranular ferrite grains on cooling. To facilitate the precipitation process of VN a sub-micro-addition of Ti was used which also helps to restrain austenite grain growth during piercing and pipe forming. Subsequent V(C,N) precipitation of the remaining vanadium in the ferrite contributes precipitation strengthening. Hot rolling followed by intragranular ferrite formation in 0.1% V-0.015% N-0.005% Ti steel is able to develop a fine ferrite-pearlite microstructure with an average ferrite grain size of 7µm.

KEYWORDS: hot rolling, pipe manufacture, microalloying, vanadium, titanium, precipitates, vanadium nitride, nucleation, ferrite, grain refinement

INTRODUCTION

Grain refinement through the formation of intragranular ferrite directly on VN that is precipitated inside the austenite grains provides an attractive new way of thermo-mechanical (TM) processing of steel products for which the usual controlled austenite conditioning is not readily applicable. Recent studies [1,2] clearly indicate that VN is the most effective precipitate for nucleation of intragranular ferrite. This is due to the fact that the atomic mismatch between VN and ferrite, along the two directions, $[001]_{VN} // [011]_{\alpha}$ and $[010]_{VN} // [01-1]_{\alpha}$ is only 2.1%, so allowing an epitaxial-type of growth of ferrite on VN edges [3]. Intragranular polygonal ferrite grains grow by migration of the incoherent boundary, involving a purely diffusional mechanism.

The formation of intragranular ferrite requires the presence of active VN particles in austenite. However, the kinetics of VN precipitation in undeformed austenite are very sluggish and require long holding times near the temperature of maximum precipitation in order to produce effective VN particles [4]. There are two ways in which the precipitation of VN in austenite can be enhanced. The first alternative is to modify

the steel composition to obtain a high density of fine particles or inclusions which may act as nucleation sites for VN, without substantially changing the processing conditions. The second alternative involves strain-induced precipitation of VN in austenite during hot rolling, in the temperature range for VN precipitation.

The specific processing conditions for seamless tubes with transfer times and intermediate reheating at temperatures suitable for VN precipitation in austenite seem to provide almost ideal conditions for intragranular nucleation. In a simplified form the technology of seamless production distinguishes three stages; (1) high-temperature piercing and hot rolling, (2) transfer employing an intermediate reheat furnace, and (3) low temperature final sizing and slow cooling. To induce intragranular nucleation in this process, several conditions must be fulfilled. The most critical involves nucleation and growth of VN in austenite. In order to facilitate this precipitation, minute additions of Ti can be used to "nucleate" VN at much higher temperatures than those for Ti-free steels. Experimental results clearly indicate that TiN particles which precipitate above 1300°C may act as nuclei, attracting co-precipitation of VN well above 950°C [5].

TiN has also a second practical significance during the high temperature processing. The presence of TiN is expected to restrain the growth of austenite grains. It was observed that in the presence of Ti, stable TiN particles restricted grain coarsening to about 40µm during severe deformation at high temperatures [6]. It was also seen that the austenite grain size is smaller for high values of VxN contents (about 30×10^{-4}) [7] so that, even in the absence of titanium, austenite grain refine-

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Steel	C	Si	Mn	Al	V	N	Ti	V(%wt)xN(%wt)
CMn	0.17	0.39	1.62	0.002	0.010	0.004	-	$0.04 \cdot 10^{-4}$
VN2	0.17	0.38	1.63	0.002	0.107	0.017	-	$18 \cdot 10^{-4}$
VN3	0.17	0.39	1.66	0.002	0.156	0.020	-	$31 \cdot 10^{-4}$
VN2Ti05	0.17	0.38	1.60	0.002	0.10	0.017	0.0059	$17 \cdot 10^{-4}$

▲
Tab. 1

Chemical composition of V-N(Ti) steels and the reference C-Mn steel.

Composizione chimica degli acciai V-N (-Ti) raffrontata con quella dell'acciaio C-Mn.

Pass No	Strain per Pass ($\ln(\epsilon_f/\epsilon_i)$)	Temperature (°C)	Delay Time between passes (s)
Reheating	1230°C		
Piercing	0.55	1215°C	
Hot rolling in 8-passes (Total red. 55%)	0.3÷0.03	1200÷1100	1÷0.5
Transfer	70 seconds from 1100°C (cooling rate ~1.5°C/s)		
Reheating furnace	In=990°C >> Out=930°C Time in furnace 12 minutes		
Sizing in 7-passes (Total red. 15%)	0.03÷0.01	930°C	~1
Cooling	~1.2°C/s to 740°C and ~0.35°C/s from 740 to 650°C		

▲
Tab. 2

L-rolling schedule with long transfer time.

Ciclo di laminazione L con tempo di trasferimento lungo.

ment is achieved with these alloy additions.

The aim of the present work was to develop a reliable technology for as-rolled seamless tubes which will eliminate the need for in-line normalising or expensive alloying. In the proposed concept of "intragranular ferrite" processing of V+N steels with sub-micro-additions of Ti, the final ferrite grain size will depend on two parameters;

- (i) the austenite grain size developed at the end of phase (1), and
- (ii) intragranular ferrite nucleation on VN particles during phase (3).

EXPERIMENTAL PROCEDURE

The composition and VxN product of the investigated steels are given in Tab. 1. The steels were produced as laboratory casts of section size 40mm x 40mm. The plain CMn steel was used a reference. The second and third were V+N steels with an increasing VxN product. The initial purpose of studying the high V+N steel was to evaluate the effect of V and N on γ coarsening resistance during the first stage of processing. As was stated above, there are indications [7] that by increasing the VxN product to about 30×10^{-4} , as in steel VN3, the size of γ grains can be significantly decreased. The last steel contains the same level of V and N as VN2 but is additionally microalloyed with 0.0059% Ti.

The specific rolling schedule of seamless tube depends primarily on the final tube size. For precipitation of VN in austenite, the most important part is the intermediate time-temperature

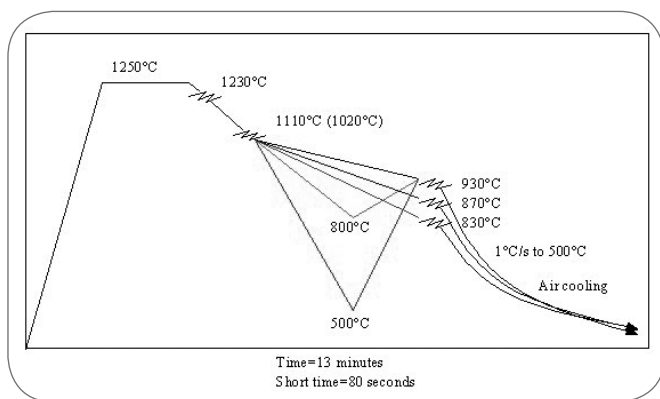
profile. Therefore, two extremes, corresponding to the long (L) transfer time and short (S) transfer time combined with different sizing temperatures have been investigated.

The base laboratory rolling variables for the L- and S-variants are shown in Tab. 2 and 3. For the L schedule the time in furnace prior to sizing was 12 minutes. For the S variant the rolling temperature was decreased to 1020°C and transfer time was limited to only 80 seconds.

Based on these processing conditions the following modifications, shown schematically in Fig. 1, were investigated. The upper part of the L-schedule was similar to that shown in Tab. 2, but the lower part, below 1110°C, was modified to examine the formation of VN in austenite. This comprised transfer to different sizing temperatures between 930°C, and 830°C with a transfer time of 12 minutes and different time-temperature profiles. In addition, cooling to 800°C prior to reheating for sizing at 930°C as well as in-line normalising with cooling to 500°C and sizing at 930°C were applied. The in-line normalising simulation was performed to obtain the reference state.

For the S-schedule the transfer time was 80 seconds from the finish rolling temperature of 1020°C. The sizing temperatures and time-temperature profiles were similar to those applied for the L-schedule. The cooling rate after sizing was in both cases 1°C/s to 500°C followed by air cooling to room temperature.

L and S rolling schedules specified in Tab. 2, 3 and Fig. 1 were reproduced in the laboratory using a Gleeble 3800 thermomechanical simulator. In such simulation, the key hot working variables are the strain, strain rate, temperature and time. This method has been employed successfully to reproduce almost exactly the whole processing of seamless tubes. Specimens were also water quenched from different stages in order to evaluate the evolution of austenite microstructure during high-temperature processing.



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Fig. 1

Schematic diagram showing rolling schedule for long transfer time (L-schedule) and short transfer time (S-schedule).

Diagramma del ciclo di laminazione per il tempo di trasferimento lungo (ciclo L) e tempo di trasferimento corto (ciclo S).

RESULTS

Austenite grain size

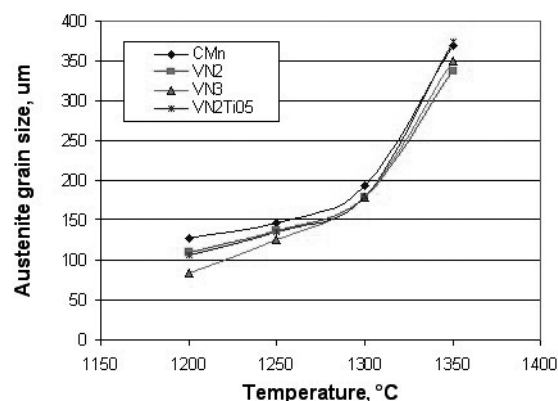
The effect of reheating temperature on austenite grain size of the investigated steels is shown in Fig. 2. It can be seen that austenite grain growth follows the same pattern for all steels with an accelerated coarsening above $\sim 1275^\circ\text{C}$. At the reheating temperature of 1250°C the average grain size was within the range 110-170 μm with the highest value for the CMn steel. An important observation was that a relatively uniform grain structure, without abnormally coarse grains, was developed in the experimental V+N and V+N(Ti) steels with high V and N contents.

The development of austenite grain size during high temperature processing is shown in Fig. 3. Piercing at 1215°C followed by hot rolling between 1200 - 1110°C produced a systematic decrease in the austenite grain size with a stronger effect for V+N steels as compared to the CMn steel. It appears from this figure that the refinement of austenite to about $60\mu\text{m}$ after hot rolling at 1110°C is due to the presence of vanadium and nitrogen in solution. The mechanism involved is not known but seems to be connected with the formation of atom pairs or clusters. This austenite refining potential of V+N, first recognised by Cryderman [7], is very significant when producing high strength seamless without a special heat-treatment.

The austenite grain refining effect was further enhanced by a small addition of Ti. For the VN2Ti05 steel the average austenite grain size after high temperature rolling was about $50\mu\text{m}$. These results indicate that a fine prior austenite grain size can be obtained after the high temperature rolling of V+N steels. The increased γ grain boundary area is very helpful in promoting ferrite grain refinement.

Contribution from intragranular nucleation to grain refinement

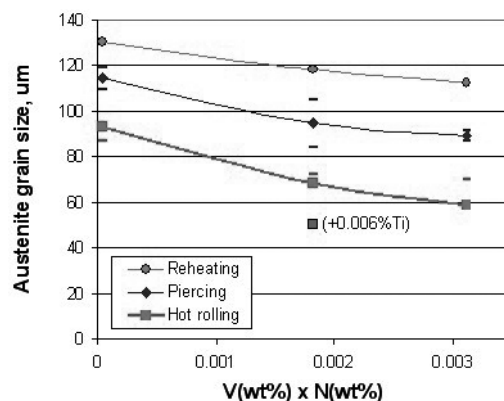
The primary parameters controlling the ferrite grain size in a traditional austenite-ferrite transformation are the effective austenite grain boundary area and the cooling rate. Once the austenite grain size is known, the resulting ferrite size can be deduced from empirical knowledge of the transformation ratio [8]. If intragranular nucleation provides additional ferrite nu-



▲
Fig. 2

Austenite grain size of V+N and V+N(Ti) steels at reheating temperatures.

Dimensione del grano austenitico negli acciai V+N e V+N(Ti) a temperature di riscaldamento.



▲
Fig. 3

Effect of V, N and Ti on austenite grain size after piercing at 1215°C and hot rolling at 1110°C .

Effetti di V, N e Ti sulla dimensione del grano austenitico dopo foratura a 1215°C e laminazione a caldo a 1110°C .

cleation sites, besides grain boundaries, the final ferrite grain size should be smaller as compared to that of the plain CMn steel. Thus the $d\gamma/d\alpha$ transformation ratio can be further used as a measure of intragranular nucleation on VN distributed inside austenitic grains.

In order to evaluate the contribution from intragranular nucleation the investigated steels were processed using the most favourable conditions for VN precipitation in austenite which comprise intermediate treatment at 800°C , within the VN precipitation curve and sizing at 930°C (Fig. 4a). All the other processing conditions were similar to that shown in Tab. 2.

The resulting microstructure for CMn, V+N and V+N(Ti) steels are shown in Figs 4(b-e). It is clearly seen from this figure that the microstructure of CMn steel is predominantly bainitic with coarse proeutectoid ferrite allotriomorphs delineating prior austenite grain boundaries. In V-steels a fine ferrite-pearlite microstructure was formed instead. An increase in ferrite

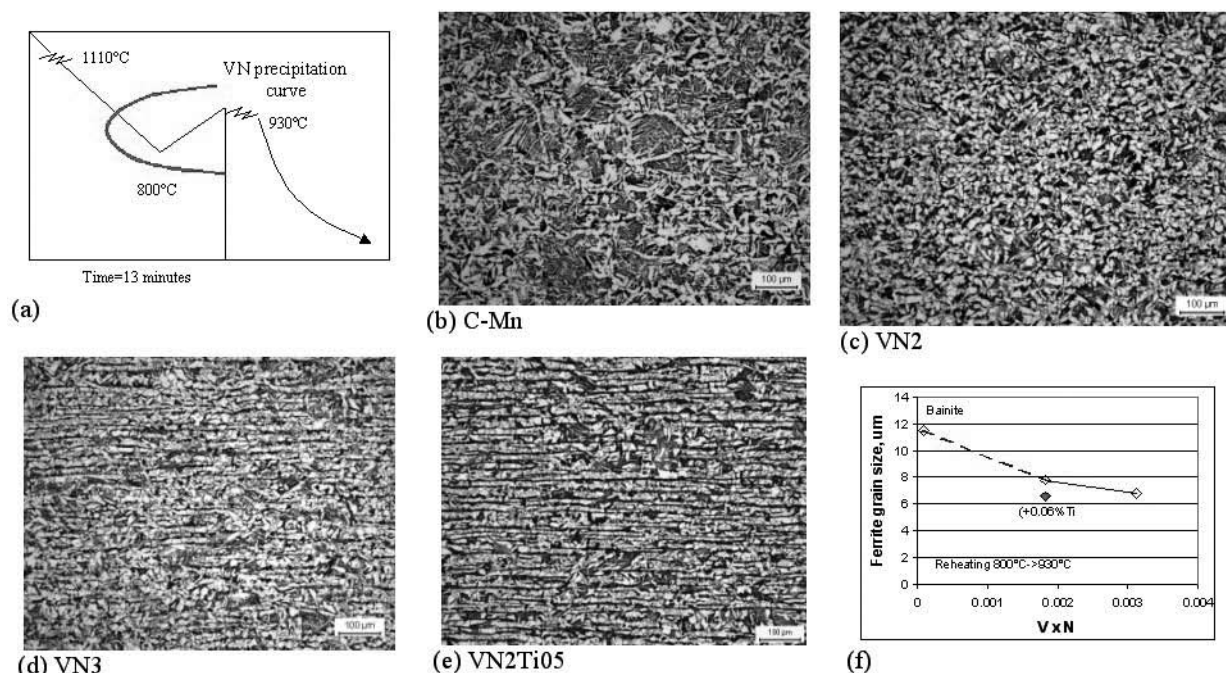


Fig. 4

Microstructures of CMn and V-steels after processing with intermediate treatment at 800°C.

Microstrutture degli acciai CMn e V dopo processo con trattamento intermedio a 800°C.

grain refinement in Steels VN3 and VN2Ti05 produced also more distinct pearlite banding. Measured ferrite grain sizes in different steels are shown in Fig. 4(f). For the V-steels the ferrite grain size was below 8 μm.

The quantitative data have been used to evaluate the transformation ratio for the V-steels, Fig. 5(a). The transformation ratio for the CMn steel is also shown in this figure. This ratio was estimated from the grain boundary ferrite and gives only a rough approximation as the interiors of austenite grains were composed of Widmanstätten ferrite and upper bainite, Fig. 5(b). Available data suggest that the transformation ratio for the normalised CMn steels with $d_{\gamma} \sim 60 \mu\text{m}$

(equivalent to the present V-steels) should be between 2 and 2.5 [8]. The transformation ratio for V-steels was ~ 8 or above depending on the initial austenite grain size. This seems to provide clear evidence that an additional nucleation mechanism, besides that at grain boundaries, is active in V-steels. The formation of fine ferritic grains inside austenite can be easily recognised in optical micrographs, as shown in Fig. 5(c). An even denser nucleation of ferrite on the prior austenite grain boundaries is also evident in this figure. This indicates that VN particles precipitate preferentially on the austenite grain boundaries in VN2 and then assist the nucleation of ferrite at these sites

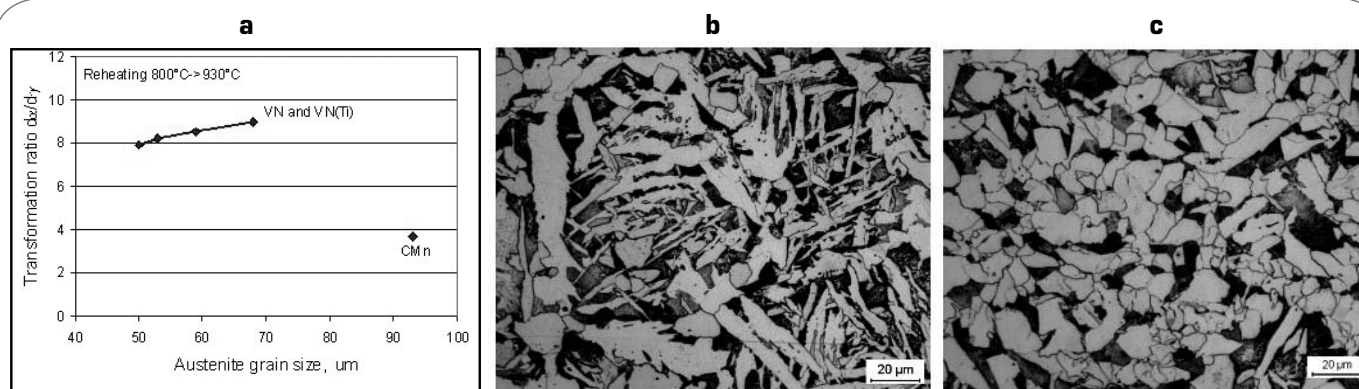


Fig. 5

Transformation ratio, d_f/d_{α} , (a) and microstructure of CMn (b) and VN2 (c) steels after processing with intermediate treatment at 800°C.

Rapporto di trasformazione, d_f/d_{α} (a), e microstruttura degli acciai CMn (b) e VN2 (c) dopo trattamento intermedio a 800°C.

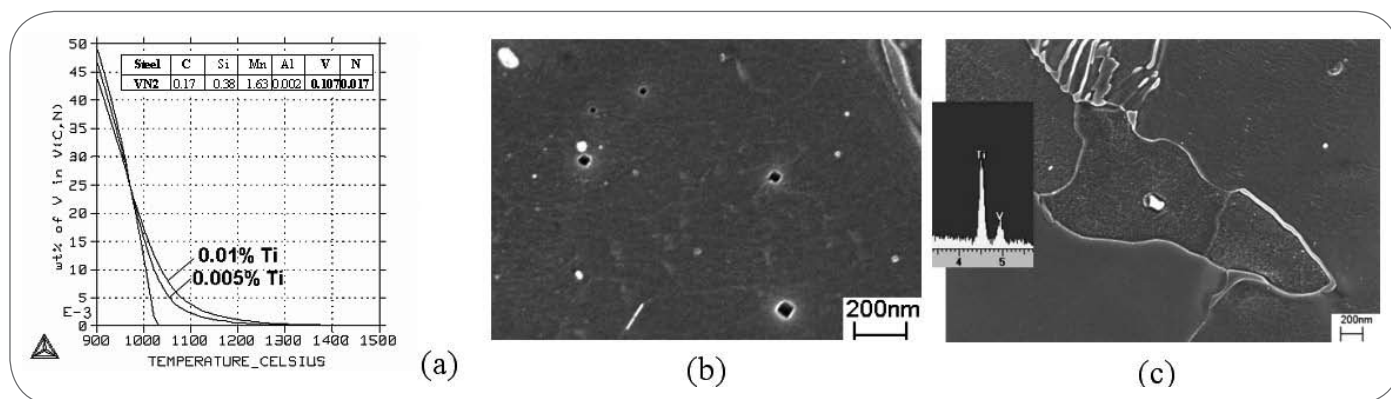


Fig. 6

Theoretical calculations of the effect of Ti on the fraction of V precipitated at equilibrium in austenite (a), and complex (TiV)N particles in steel VN2Ti05 (b).

Calcoli teorici dell'effetto del Ti sulla frazione di V precipitato in condizioni di equilibrio nell'austenite (a), e sulle particelle del complesso di (TiV)N nell'acciaio VN2Ti05 (b).

Effect of small titanium additions

Many experimental studies have shown that VN may co-precipitate to a large extent on already existing TiN-particles. Fig. 6 shows the effect of Ti on the fraction of V precipitated at equilibrium in austenite, calculated from the thermodynamic database for microalloyed steels. It can be seen that the temperature for initial precipitation of VN is increased in Ti-steels. The individual particles will thus grow as (Ti,V)N with V-rich surface layers. In accordance with this reasoning, larger particles should be found in Ti-steels whereas smaller ones should be formed in Ti-free steels. Furthermore, it may be expected that as long as V co-precipitates on pre-existing TiN as the temperature decreases, the density of precipitates will be different for VN and VN(Ti) steels.

The conceptual effect of Ti on the shift of the C-curve for VN precipitation to higher temperature and shorter holding times is shown schematically in Fig. 7(a) and confirmed by microstructure investigation of Ti free and Ti bearing steels, Fig. 7(b,c). If there are no effective particles, due to the lower stability of VN in the Ti-free steel VN2, the increased hardenability causes the formation of Widmanstätten ferrite and upper

bainite structures within austenitic grains, (Fig. 7(b)). When the C-curve was shifted to higher temperature by a small addition of Ti, decoration of TiN by VN took place, and a fine ferrite-pearlite microstructure developed instead after sizing at 930°C. These results clearly illustrate that the formation of intragranular ferrite in V-steels is significantly enhanced by small additions of Ti which increases the stability of VN in austenite.

Effect of sizing temperature

The sizing temperature is one of the most important parameters for intragranular nucleation as it determines the conditions for VN precipitation during intermediate treatment. In the present simulations the sizing temperature was varied between 930°C-830°C after hot rolling at 1110°C and transfer time of 13 minutes. The effect of sizing temperature on the final microstructure of VN and VN(Ti) steels is shown in Fig. 8.

A systematic change in the microstructure with the sizing temperature and composition can be seen from this figure. The Ti-free steel VN2 was not able to develop ferrite-pearlite microstructure after sizing at the high temperature of 930°C. Even sizing at 870°C produced a partially bainitic microstructure.

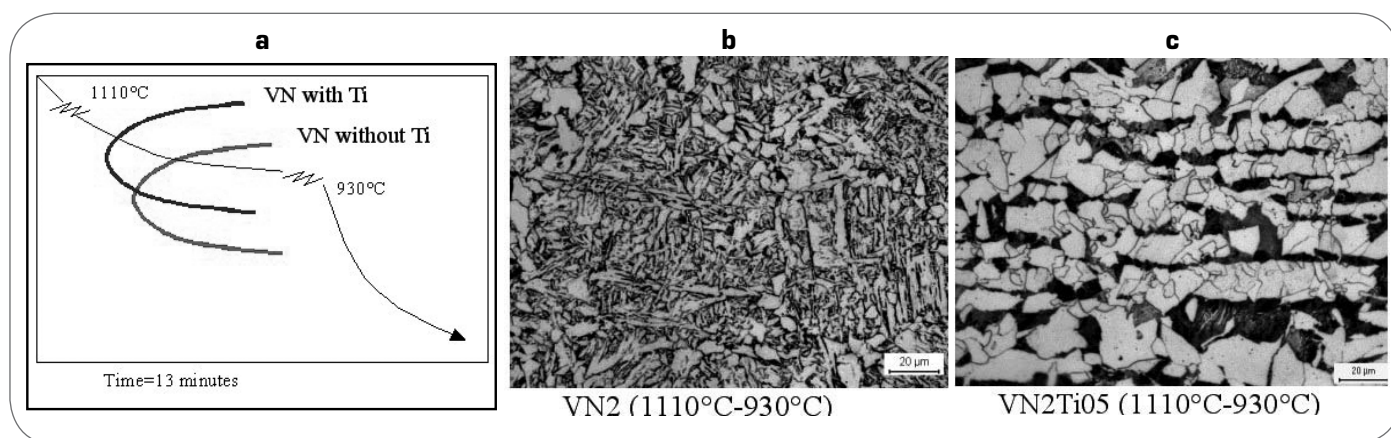


Fig. 7

The effect of Ti on the shift of the VN C-curve (a) and the microstructure of VN2 (b) and VN2Ti05 (c) steels after sizing at 930°C.

Effetto del Ti nella transizione della curva VN C (a) e microstruttura degli acciai VN2 (b) e VN2Ti05 (c) dopo calibratura a 930°C.

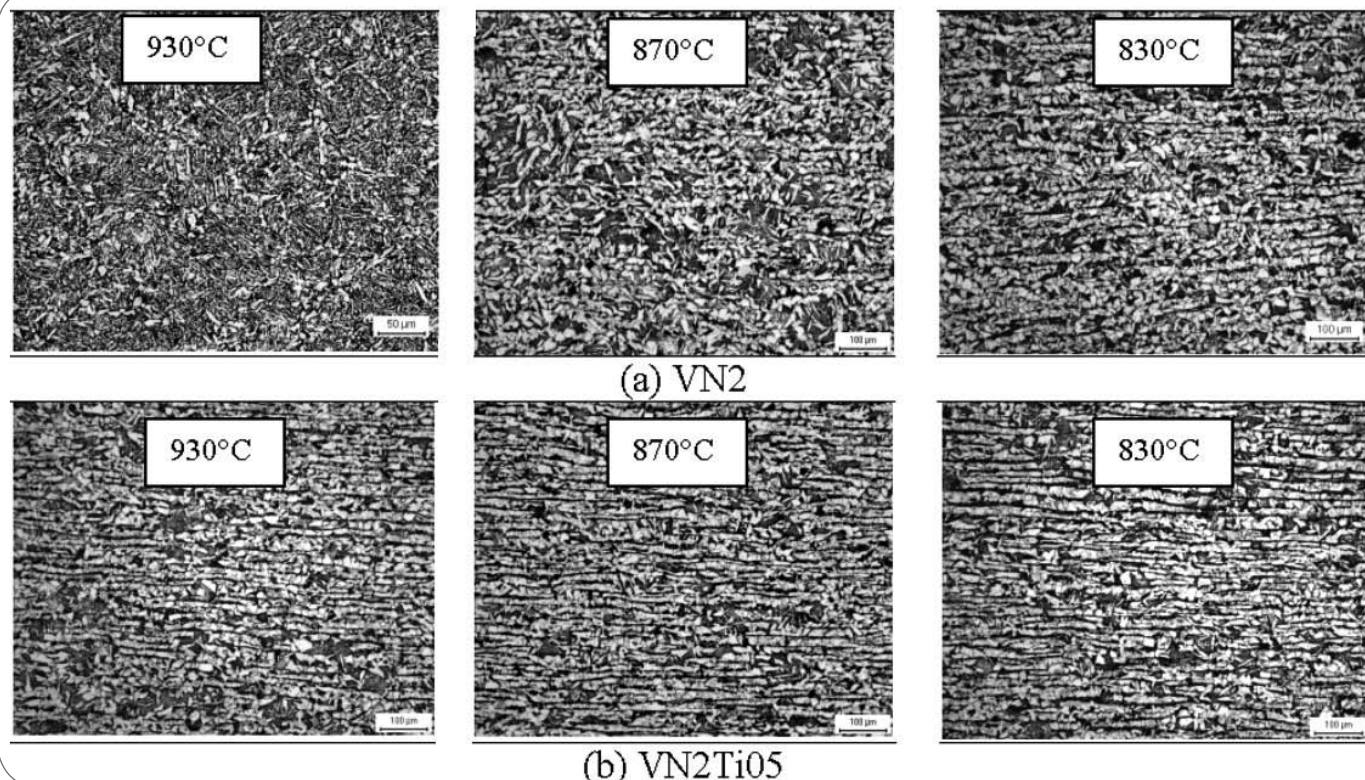


Fig. 8

The effect of sizing temperature on final microstructure in steels VN2(a) and VN2Ti05 (b).

Effetto della temperatura di calibratura sulla microstruttura finale negli acciai VN2(a) e VN2Ti05 (b).

This microstructure can be improved by microalloying with higher V+N, as in steel VN3, or by further decreasing of the sizing temperature. The best solution, according to the present results, is offered by a small addition of Ti to V-steels. In the presence of Ti sizing at 930°C was able to produce good ferrite-pearlite microstructure. Further analysis of the ferrite grain size is given in Fig. 9(a), in which the area of bainite formation is also indicated. Comparing Ti-free and Ti-bearing steels it can be seen that in the presence of Ti the sizing temperature

is less critical for precipitation of VN and intragranular ferrite formation.

Short transfer time

Depending on the final tube size the specific rolling schedule may also involve fast processing. In this case the time for precipitation is reduced and active VN particles may not develop. Therefore the second case investigated in this work comprised processing with a short transfer time of 80 seconds. The micro-

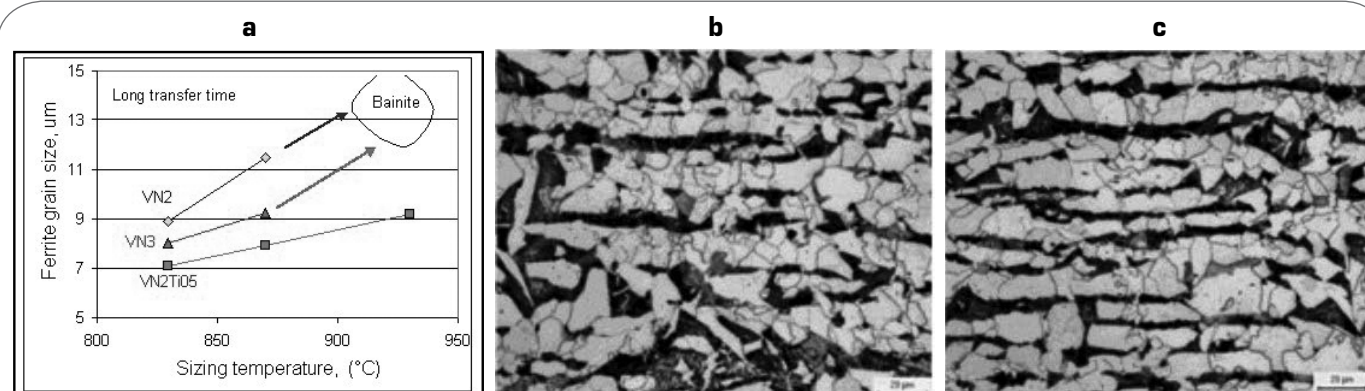
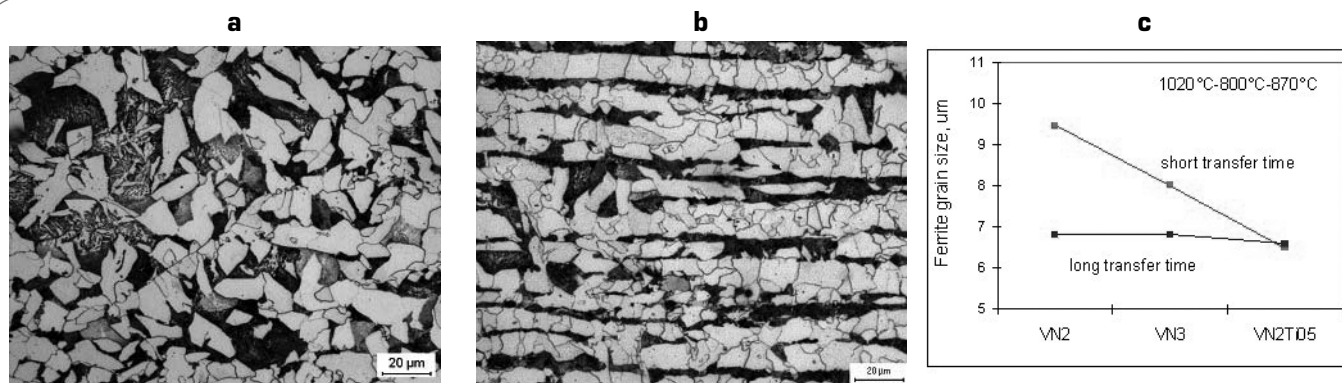


Fig. 9

The effect of sizing temperature on ferrite grain size (a) and the microstructure of VN2Ti05 steel at 930°C (b) and 830°C (c).

Effetto della temperatura di calibratura sulla dimensione del grano di ferrite (a) e sulla microstruttura del grano dell'acciaio VN2Ti05 a 930°C (b) e 830°C (c).



▲
Fig. 10

Final microstructures of VN2 (a) and VN2Ti05 (b) and ferrite grain size (c) after processing with the short transfer time.

Microstrutture finali degli acciai VN2 (a) e VN2Ti05 (b) e dimensione del grano ferritico (c) dopo lavorazione con un ciclo di trasferimento corto.

structure after S treatment with 800°C intermediate temperature and sizing at 870°C is shown in Fig. 10. It was found that the Ti-free steels cannot be processed with short transfer time as intragranular ferrite cannot be fully developed. Furthermore, it can be seen in Fig. 10(c) that Ti-steel VN2Ti05 exhibited the same ferrite grain size after short and long intermediate treatment. It must, however, be pointed out that sizing temperature should be limited to 870°C as bainite formation was observed at 930°C.

Comparison of microstructures from intragranular nucleation with in-line normalising

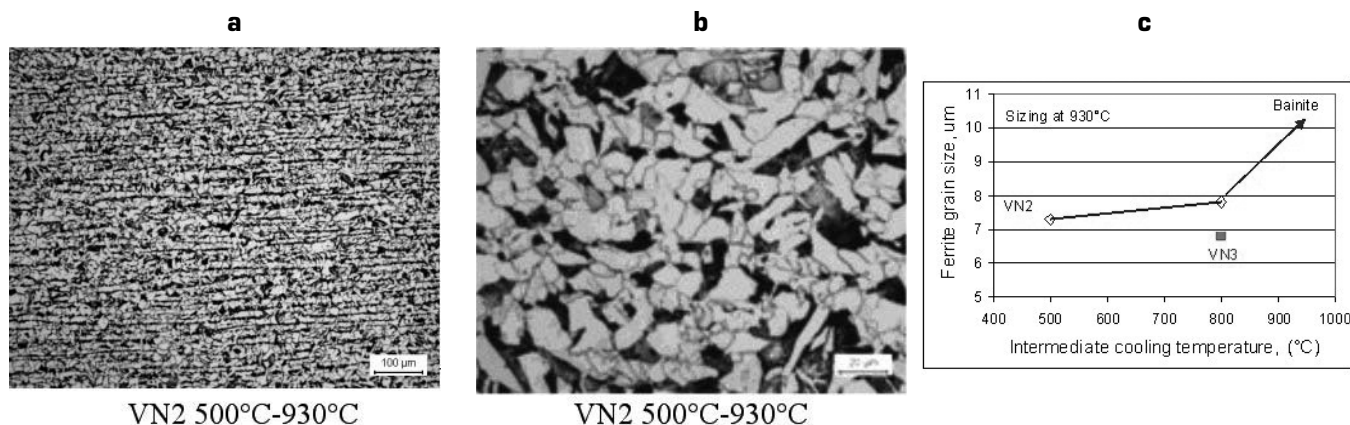
In-line normalising is used to provide refinement of the austenite grains prior to transformation for ensuring toughness. Grain control during normalising in the V-steels is a result of the precipitation of VN. When V is added to the steel, the austenite grain size developed at common normalising temperatures up to about 930°C is considerably less than that observed in plain carbon steels due to the austenite grain growth inhibiting effect of V carbonitrides and this assists refinement of the ferrite grains during transformation from the austenite on

cooling.

In-line normalising was simulated in the present case on the VN2 steel by cooling to 500°C to assure full transformation to ferrite before reheating to the sizing temperature of 930°C. The resulting microstructure is shown in Fig. 11. The results from quantitative measurements indicate that the ferrite grain size after in-line normalising was similar to that obtained from intragranular nucleation. Based on these results it can be concluded that intragranular nucleation is able to replace in-line normalising.

Nucleation of intragranular ferrite

The microstructure investigation revealed that two types of ferrite nucleation sites are active in V-steels. The first one are the austenite grain boundaries and the second are in the grain interiors. Fig. 12(a) shows a profound nucleation of ferrite on the prior austenite grain boundaries. It appears that the austenite grain boundaries are the preferential precipitation site for VN nucleation in Ti-free steels as homogeneous nucleation within grains requires higher driving forces. The situation is different in Ti-bearing steels as VN can grow epitaxially on the



▲
Fig. 11

Final microstructures (a) and (b) and ferrite grain sizes (c) after processing with in-line normalising.

Microstrutture finali (a) e (b) e dimensione del grano ferritico (c) dopo lavorazione con normalizzazione in linea.

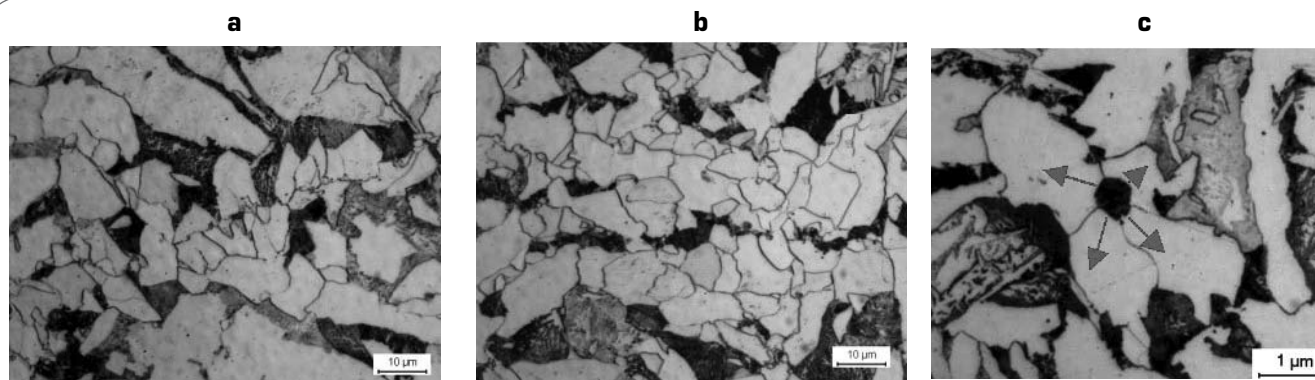


Fig. 12

Abundant ferrite nucleation from grain boundaries in VN2 (a), in the interior of austenite grains in VN2Ti05 (b) and an example of four-arm ferrite rosette growing from one particle in (c).

Abbondante nucleazione della ferrite dai bordi del grano nell'acciaio VN2 (a), e all'interno dei grani di austenite nell'acciaio VN2Ti05 (b) e un esempio di rosetta ferritica a quattro bracci cresciuta da una particella in (c).

existing TiN which themselves had nucleated inside the austenite grains previously, so giving more uniform grain structure, Fig. 12(b).

In this case the nucleation is facilitated and obviously requires less energy. The best verification of intragranular nucleation is the observation of four-arm ferrite rosettes at one particle, as shown in Fig. 12(c). Such rosettes have been observed previously [1] when the mechanism of intragranular ferrite formation on VN was investigated.

Hardness

Hardness measurements have been performed to estimate the possibility for additional precipitation strengthening of ferrite. According to thermodynamic calculations the VN precipitation in austenite involves only a part of vanadium leaving the rest available for precipitation in ferrite. Slow cooling may be advantageous in this respect for the formation of V(C,N) in ferrite. V-steels exhibited much higher hardness as compared to that of the plain C-Mn steel and their hardness level was dependent on the type of intermediate treatment, Fig. 13. The highest

hardness was obtained after sizing at 870°C. The hardness of Ti bearing VN2Ti05 steel was lower due to the higher fraction of V tied up in austenite in the presence of TiN. These observations are consistent with the microstructural findings and indicate that V can be used simultaneously for (i) providing grain refinements and (ii) for precipitation strengthening which is the most effective way of using microalloying.

DISCUSSION

Traditional manufacturing cycles for seamless tube provide materials with high-tensile properties but are not always able to guarantee good transversal toughness at low temperatures. This is directly connected with the fact that piercing and hot rolling must be performed at high temperatures during which the optimum control of austenite grain size is not possible. The resulting coarse ferrite grain structure may show inadequate toughness. In-line normalising treatments must then be used to refine the austenite and subsequent ferrite structures. However, in-line normalising is not favourable from an economic

point of view and may also be questioned technically. If, for example, austenite is not completely transformed during the normalising heat treatment then persistent high carbon austenitic islands may form and deteriorate the final microstructure.

To overcome these difficulties an alternative approach to grain refinement through intragranular ferrite formation on VN is proposed in this work. This approach is based on utilising the transfer/heating time between hot rolling and sizing for precipitation of VN particles in austenite which in turn nucleate ferrite on cooling. To facilitate the precipitation process of VN, traces of Ti are used which helps to "nucleate" VN at higher temperatures by co-precipitation on the existing TiN particles. This provides a robust microalloying

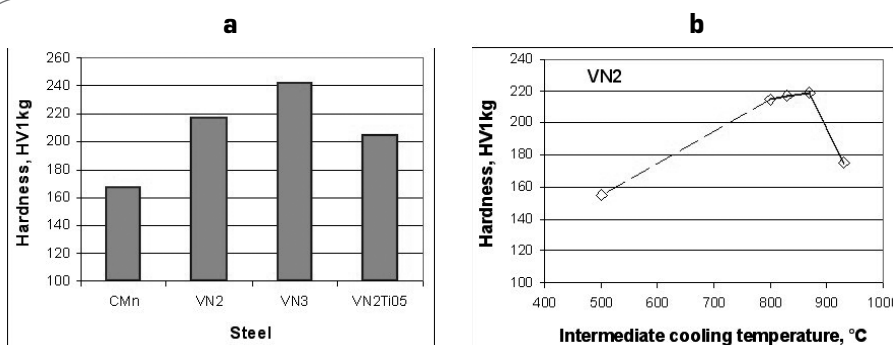


Fig. 13

Hardness as a function of steel chemistry (a) and intermediate cooling temperature (b).

Durezza in funzione della composizione dell'acciaio (a) e della temperatura di raffreddamento intermedio (b).

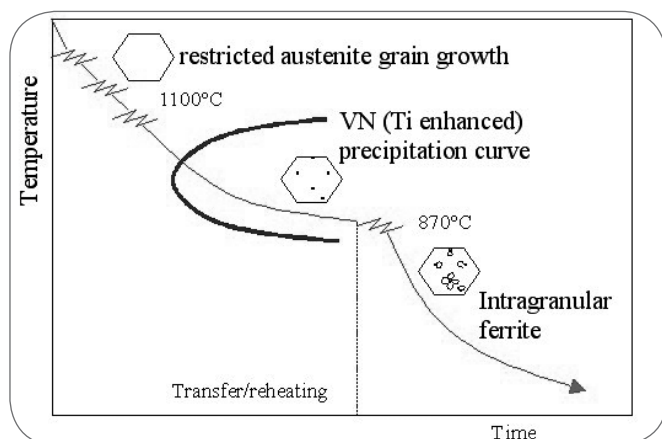


Fig. 14

Schematic diagram showing processing of V+N(Ti) steel with intragranular ferrite formation.
 Diagramma schematico della lavorazione dell'acciaio V+N(Ti) con formazione intragranulare di ferrite.

system able to develop active VN, both during processing with short transfer time and/or sizing at higher temperatures.

In the V+N(Ti) system, TiN contributes to the restriction of austenite grain growth during piercing and pipe forming while the yield strength can be further enhanced by precipitation hardening from vanadium that remains in solid solution after precipitation in austenite. Based on these assumptions a new process with intragranular nucleation, shown schematically in Fig. 14, has been proposed. Control of austenite grain growth during piercing and pipe forming together with VN formation during transfer/heating for nucleation of intragranular ferrite are used to refine the final microstructure after processing and eliminate the necessity of in-line normalising or any treatment during sizing.

The potential of grain refinement through the formation of intragranular ferrite on VN can be estimated using the d_f/d_a transformation ratio. Fig. 15 summarises the experimental data for the present V+N and V+N(Ti) steels and compares these with normalised CMn steels. For the range of austenite grain sizes investigated in this work, intragranular nucleation provided an additional refinement which was at least 2.5 times greater than that from austenite grain boundaries.

The role of titanium

It is well established that TiN forms as a fine precipitate dispersion in the early stage after solidification in microalloyed steels having less than about 0.015%Ti. At lower temperatures when the solubility of V(C,N) has been exceeded many experimental studies have shown that this phase co-precipitates on already existing TiN-particles to a large extent. There is evidence for this behaviour of the V-Ti-N system both in the thermodynamics and in the fact that Ti-rich (VTi)N coexists with V-rich (VTi)N at high temperatures where interdiffusion should lead to homogenisation. Based on these results, the concept of a miscibility gap in the V-Ti-N system was presented such that there is de-mixing to Ti-rich (TiV)N core and V-rich (TiV)N surface layers [5]. In accordance with this reasoning it was found that V-rich surface zones were formed on fine, cube-like TiN. The most important consequence of the presence of Ti is that the nose of VN precipitation reaction is shifted to higher temperatures and shorter times.

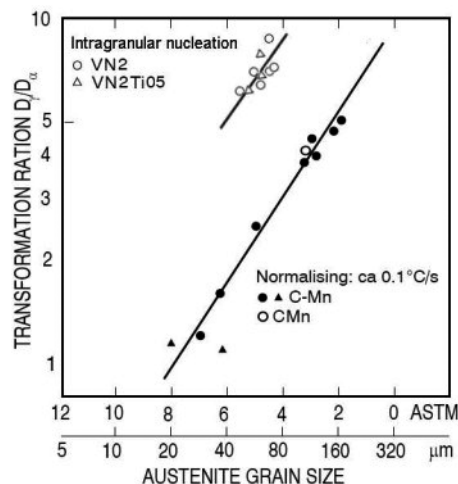


Fig. 15

The d_f/d_a transformation ratio for intragranular nucleation.

Rapporto di trasformazione d_f/d_a per la nucleazione intragranulare.

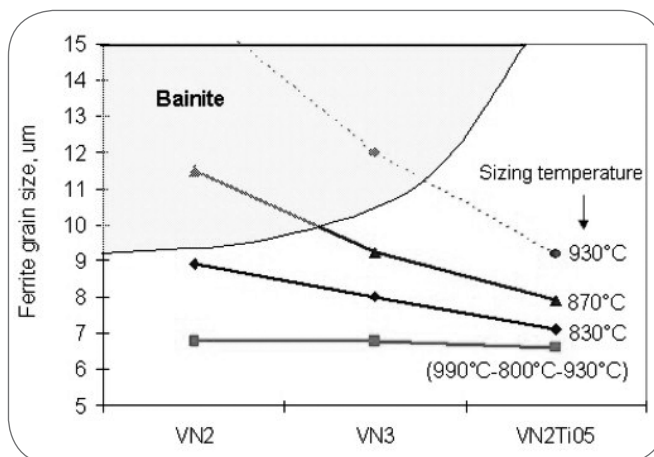


Fig. 16

The effect of sizing temperature on the ferrite grain size for V+N and V+N(Ti) steels.

Effetto della temperatura di calibrazione sulla dimensione del grano ferritico per gli acciai V+N e V+N(Ti).

Sizing temperature

It was noted previously in mill trials that the sizing temperature may affect impact toughness. The best results were obtained using temperature slightly below 900°C. It has been speculated that variation in toughness are caused by differences in ferrite grain size which may reflect different degrees of intragranular nucleation. The present results seem to confirm this behaviour and provide a reliable explanation for the formation of mixed microstructures. The conditions for intragranular nucleation on VN depend on the time-temperature profile between hot rolling and sizing. This is clearly seen in Fig. 16. If sizing is performed at high temperature relative to the C-curve for VN precipitation, then VN particles are not able to fully develop in the centre of austenite grains, leading to a mixed microstructure with Widmanstätten ferrite and bainite. In the presence of Ti the sizing temperature is less critical as the bainitic component

can be avoided at all sizing temperatures. Therefore, it can be concluded that traces of Ti are indispensable for providing the robust microalloying system with V+N that is insensitive to variations in the sizing temperature or transfer time.

CONCLUSIONS

The new mechanism concept of grain refinement through the formation of intragranular ferrite on VN has been applied to seamless pipe production. The major conclusions of this investigation are the following:

1. Standard processing of V-steel with 0.1%V-0.015%N is able to induce intragranular ferrite and produce a fine ferrite-pearlite microstructure with an average ferrite grain size of 7µm.
2. In this processing transfer/heating time between hot rolling and sizing is used to precipitate VN in austenite which in turn is used to nucleate intragranular ferrite grains.
3. Sub-micro-additions of Ti are used in this composition to facilitate the precipitation of VN in austenite on the existing TiN particles, enabling development of active VN particles, during processing with short transfer time or sizing at higher temperatures.
4. In the V+N(Ti) system, TiN is also useful for restricting austenite grain growth during piercing and pipe forming and the yield strength is further enhanced by precipitation strengthening

from the vanadium remaining in solid solution after precipitation in austenite.

5. The presence of Ti makes the rolling process less critical with respect to the sizing temperature as the bainitic components may be avoided even after sizing at relatively high temperatures.

6. The proposed concept of rolling with intragranular ferrite formation is able to eliminate in-line normalising, and thus provides a clear economical benefit for seamless tube processing.

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ABSTRACT

AFFINAZIONE DEL GRANO FERRITICO IN TUBI SENZA SALDATURA MEDIANTE NUCLEAZIONE INTRAGRANULARE SU VN

Parole chiave: acciaio, laminazione a caldo, fabbricazione, microalloying, vanadio, titanio, nucleazione

Nel presente studio è stato simulato il processo di laminazione di tubi senza saldatura con l'obiettivo di ottenere l'affinazione del grano mediante la formazione di ferrite intragranulare su VN precipitato all'interno di grani austenitici, per la produzione di microstrutture come da laminazione a caldo, che attualmente possono essere solo ottenute mediante normalizzazione in linea. Le prove sono state effettuate su acciai V + N e V + N (Ti) utilizzando due tipi di ciclo

con tempi di trasferimento/riscaldamento lunghi e brevi prima della calibratura tra 930 e 830° C. Si è rilevato che il tempo di trasferimento/riscaldamento tra la laminazione a temperatura elevata e la calibratura a bassa temperatura poteva essere utilizzato per la precipitazione del VN, entro l'austenite, che a sua volta poteva nucleare grani di ferrite intragranulari nel raffreddamento. Per facilitare il processo di precipitazione del VN è stata utilizzata anche una sub-micro-aggiunta di Ti che ha contribuito a limitare la crescita del grano austenitico durante la foratura e la formatura del tubo. La successiva precipitazione di V (C, N), vanadio rimanente nella ferrite contribuisce all'aumento di resistenza meccanica da precipitazione. Il processo di laminazione a caldo seguito dalla formazione intragranulare della ferrite nell'acciaio 0,1% V-N 0,015% 0,005%-Ti in è in grado di sviluppare una microstruttura fine di ferrite-perlite, con una dimensione media del grano di ferrite di 7µm.