# NIOBIUM AND HEAT AFFECTED ZONE MYTHOLOGY

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# Abstract

The myth that niobium levels should be restricted in steel specifications for structural and line pipe applications, to avoid unacceptably low HAZ toughness, is considered herein and effectively dispelled. Furthermore it is demonstrated that control of carbon level and the judicious use of primary alloying elements should always allow steelmakers to identify attractive compositional options which more effectively utilise the unique properties of niobium to provide optimum combinations of plate and HAZ mechanical properties. The latter objective becomes possible because the important factors that control HAZ toughness in HSLA steels microalloyed with niobium are now more thoroughly appreciated; this should ensure that steels supplied for individual projects are metallurgically tailored to be compatible with the welding processes appropriate to the envisaged application.

# Introduction

Niobium, the focus of my attention in this paper, has always been an element shrouded in mystery. Originally discovered and named columbium in 1801 by Charles Hatchett [1], an eminent Fellow of the Royal Society, it was for many years indistinguishable from tantalum until in 1844 a German chemist, Heinrich Rose [2], was able to demonstrate subtle differences between the two elements with reference to available valence states. Rose, appropriately, renamed Hatchett's element niobium and those of you familiar with Greek Mythology will appreciate that Niobe was a daughter of Tantalus, king of Sipylos. Tantalus, who was uniquely favoured amongst mortals by being invited to share the food of the Gods, was in turn a son of Zeus the supreme ruler of all the Gods.

With this intriguing background and such an illustrious lineage it is, perhaps, not too surprising that niobium has become a microalloying element of particular distinction and that its usage in steelmaking has from time to time given birth to modern day myths some of which have been difficult to dispel. Niobium was first identified as being beneficial to the strength of hot rolled carbon-manganese steels in the late 1930s [3] and, while the precise strengthening mechanism was not fully understood at the time, it was believed to be a consequence of the grain refining effect of niobium carbides and/or nitrides. However, it was considerably later before the use of niobium became widespread in a variety of steel products and Gray [4] has recently reminded us that its first use in linepipe occurred in 1959. We are also reminded by Gray & Siciliano, in a comprehensive historical review [5], that the introduction of niobium was not without its challenges. Initially it was noted that, in some circumstances, lower plate Charpy-notch toughness could be recorded. This was first attributed to the presence of Widmanstatten ferrite or

cementite networks on ferrite boundaries [6, 7] and later also to precipitation hardening by niobium carbonitrides [8]. Fortunately it was soon realised these problems could be overcome simply by increasing manganese content [9] and modifying rolling schedules to achieve fine ferrite grain sizes.

# There is a salutary lesson here for all of us when we turn our attention to the Heat Affected Zone.

Indeed niobium has confounded its critics and now, either alone or in combination with other microalloying elements, has become a virtually irreplaceable constituent of a wide range of modern higher strength steels for applications across the full spectrum encompassing automotive, wires & rods, structural steels for buildings, the offshore sector, bridges, cranes, ship plate, LPG containment and not least, linepipe steels for the increasingly demanding applications now being encountered.

However, we should remind ourselves that niobium's unique capability is to dramatically improve parent material properties and it is never added to steel with a view to improving either weld metal or heat affected zone toughness.

In the 1970's, when the majority of the parent material properties were adequately understood, attention inevitably focused on the role of niobium in the HAZ. To a degree this was driven by the introduction of copious new specifications for weldments in the offshore and linepipe sectors and it wasn't long before conflicting observations in the literature caused those concerned with material specifications to develop reservations concerning the value of niobium as a microalloying element. Arbitrary specification limits were sometimes introduced without sound technical justification and it was clearly necessary for the subject to be carefully examined.

In 1980, a comprehensive review of the role of microalloying elements in controlling HAZ toughness was undertaken [10] and this addressed, in some detail, the specific behaviour of niobium and how this important element could be harnessed to best effect. The review concluded that HAZ toughness is primarily controlled by microstructure and that the most important factor in this context is transformation temperature. Clearly the latter is, in turn, controlled by, inter alia, composition and cooling rate through the critical temperature range associated with the transformation of austenite to lower temperature products. The review further demonstrated that, in this context, elements such as niobium are no less and no more important than carbon or other primary alloying elements such as manganese, silicon, molybdenum or nickel. By comparing the roles of niobium at different carbon levels and over a wide range of cooling rates it was demonstrated that the optimum use of this key element is facilitated when its incorporation into steel is an integral part of a carefully conceived approach to alloy design taking full account of the end application and the welding processes envisaged for use in practice.

Of course, the latter review dealt almost exclusively with Coarse Grained HAZ (CGHAZ) toughness because in the era of higher carbon steels it was inevitably that zone which exhibited the lowest toughness and more importantly the zone in which the most common welding defects, such as hydrogen induced cold cracking, were to be found. Now, in spite of decades of successful progress with niobium containing microalloyed steels, some thirty years later it has become necessary to revisit the subject for the following reasons. Myths are by their very nature stubborn to dispel and in recent years new, and potentially valuable, contributions to the

literature have, to a degree, unwittingly reawakened concern about the specific role of niobium both in the CGHAZ and, in multi pass welding, in the Inter Critical Heat Affected Zone (ICHAZ) of HSLA steels. The latter zone is more specifically defined in the following section and referred to, as appropriate later in this paper.

#### So where are we now and how did we get here?

# The Heat Affected Zone

When steels are welded there is inevitably an area of adjacent original plate material where the microstructure and mechanical properties are significantly altered by the thermal cycles associated with the welding process in question. Many authors have very adequately described the nature and extent of this important region, or heat affected zone (HAZ) as it is most widely referred to, but for my present purposes I have elected to rely on Figure 1, below, as originally published by Batte et al [11]. This useful diagram pinpoints the location and origin of the most important subzones of the heat affected region in both two pass and multi pass welds and I propose to employ the nomenclature of Figure 1 when describing zones of particular interest in subsequent sections of this paper.



Figure 1. Heat affected zone microstructural development. After Batte et al [11].

I have already indicated my intention to concentrate my observations on the two zones widely accepted to be of most significance in weldments in HSLA steels. The coarse grained heat affected zone (CGHAZ) is the region immediately adjacent to the weld itself in which the steel has effectively been re-austenitised prior to re-transforming during cooling to a variety of lower temperature transformation products depending on composition, heating and cooling rates. The inter-critically reheated grain coarsened zone (ICHAZ) is that part of an original CGHAZ where the heating cycle of a subsequent weld pass results in partial transformation to austenite. As described by Batte et al [11] this particular effect, which is manifested in very localised areas of typical weld HAZ's, results in an increase in the fraction and size of small pockets of carbonenriched austenite the transformation behavior of which will be more fully discussed later. Clearly quite complex microstructural mixes can be expected to result from the subsequent transformation of such partially re-austenitised material but the specific effects will be dictated by composition and weld thermal cycles. The heating rate, maximum temperature and dwell time in the austenitic region are clearly of interest and these vary significantly from one welding process to another. These variants are of particular importance in steels containing microalloying elements such as niobium, vanadium or titanium and will be alluded to later. However it is the cooling rate associated with any particular thermal cycle that is of greatest interest as this is the principal factor controlling transformed microstructure and therefore toughness.

It has become common practice in studies of the HAZ to report on either the cooling rate through the critical temperature range 800 °C to 500 °C or more specifically on the time taken to cool between these two temperatures. In most papers this latter parameter is designated as  $\Delta t(800-500)$ . In practice this cooling time is a complex function of plate thickness, any pre-heat prior to welding and of course the heat input of the welding process being applied. In multi pass welds the retained heat from the previous pass must also be accounted for.

The complex relationships are most accurately described by formulae derived from Rosenthal's heat flow equations [12] and diagrams similar to that in Figure 2 below for thinner plate situations have been incorporated into appropriate European and British standards [13] to provide guidance on the  $\Delta t(800-500)$  values which might be expected in practical welding situations over a range of typical heat inputs. Such diagrams must be carefully consulted and it is important to study the source document [13] diligently to ensure that the heat flow regime (2D or 3D) has been correctly assessed.



Figure 2. Cooling times for two and three dimensional heat flow as a function of heat input (no pre-heat). Demonstrates variation with plate thickness. Adapted from BS EN 1011-2[13].

For the purposes of this paper it suffices to set out the range of  $\Delta t(800-500)$  figures that could be encountered in a range of structural and linepipe steels during their fabrication. Very low heat inputs, 0.5 to 1.5 kJ/mm, associated with Manual Metal Arc Welding or typical single arc pipe girth welding processes will, even in thin plates, result in  $\Delta t(800-500)$  cooling times of less than 15 seconds. In thicker plates, 25-50 mm, cooling times will be at the lower end of the spectrum, 3-7 secs, because 3D heat flow is involved. Submerged arc welding of pipeline steels up to 20 mm thick with twin arc heat inputs in the range 3.5 to 6 kJ/mm might produce  $\Delta t(800-500)$ times between 35 and 80 seconds and electroslag welding of plates above 25 mm with heat inputs of >25 kJ/mm will result in very slow cooling rates with  $\Delta t(800-500)$  times of >200 seconds. It is useful to have a feel for these various figures when comparing data from one technical contribution to another and also to ensure that the conclusions being drawn are practically relevant to the application envisaged.

As already alluded to, in multi pass welds or, for example, in dual torch girth welding care must be taken to account for the heat retained in the plate or pipe from an earlier pass. Figure 3, adapted from Poole et al [14] illustrates the potential significance of this factor by comparing single and dual torch welding of 16 mm thick X80 pipe and it is clear that the latter significantly alters the effective thermal cycle. This has obvious consequences for the development of transformed microstructure and for the mechanical properties of the HAZ. The significance of this factor has been further emphasised and discussed for pulsed GMAW dual torch girth welding by Chen et al [15] and for four wire submerged arc seam welding by Moeinifar et al [16] who recorded effective heat inputs of up to 8 kJ/mm in 17.5 mm linepipe (resulting in much slower cooling rates than might have been expected).



Figure 3. A comparison of single and dual torch welding on mid thickness thermal history during girth welding of 16 mm thick X80 linepipe. After Poole et al [14].

Heat affected zones are typically only a few millimetres wide and Easterling [17] has noted that their visible (etched) boundaries, in transformable steels, correspond to the peritectic temperature on the weld side and Ac1 (the lower critical temperature upon heating) on the unaffected base metal side. Poorhaydari et al [18] have demonstrated that HAZ widths might vary from around 1.86 mm at a heat input of 0.5 kJ/mm to 7.4 mm at 2.5 kJ/mm. Clearly very high heat inputs from multi arc systems or processes such as electro-slag welding would result in significantly wider HAZs.

The restricted dimensions of HAZs raise many issues when selecting a suitable test procedure to assess toughness as discussed in a later section of this paper. However, for this reason, many systematic studies on the roles of individual elements or combinations of elements employ weld thermal simulation techniques using a Gleeble and/or an alternative high speed dilatometric technique which can provide information on transformation start and finish temperatures. Furthermore, in real welds, it is often difficult to appreciate the full effects of steel processing and compositional variables on transformation behavior and microstructure and weld simulators provide an inexpensive, simple and rapid method of obtaining bulk homogeneous samples which can be used for microstructural examination and mechanical testing. Such techniques have their critics because it is claimed that test pieces derived from simulated material do not properly recreate/duplicate the metallurgical behavior or geometric restraint of the small regions of real welde joints. This is of course true but, as a comparison tool, they provide an invaluable aid to the understanding of the metallurgical effects of compositional variations and cooling rates on discrete weld regions.

In my personal opinion some of the most comprehensive and informative studies of factors affecting HAZ microstructure and toughness have very successfully employed the simulation technique and I make no apologies for relying heavily on such data in developing my arguments and theories in the following sections of this paper.

# The Coarse Grained Heat Affected Zone

Before turning our attention to the role of niobium it is necessary to understand the possible effects of the primary alloying elements identified earlier. There are a great many published papers that address this topic but I have chosen only a few which I feel best illustrate the key lessons which need to be absorbed before we can hope to understand the more complex possibilities which arise when niobium is introduced into the system.

In the mid 1970s, Bufalini et al [19] at CSM Italsider completed a comprehensive study of the effects of carbon, manganese, nickel and molybdenum on CGHAZ toughness. Their work, which was conducted using the Gleeble simulation technique, was part of a broader programme attempting to identify preferred routes for the manufacture of X70 and X80 pipeline steels. In this context strength, toughness and general weldability were all equally important. Two nominal carbon levels (0.06% and 0.12%) and three manganese levels (1%, 1.5% and 2.0%) were incorporated into the study. Nickel levels of 0.75 and 1.5 percent and molybdenum levels of 0.35 and 0.7 percent were also woven into a systematic study involving 54 experimental heats. Figures 4a, 4b and 4c reveal a few interesting findings from their work and will, I hope, serve to challenge the conventional wisdom in this area of welding technology.



Figure 4. The effects of manganese (C = 0.12%), nickel (C = 0.12%, Mn = 1.5%) and carbon (Mn = 1.0%) on CGHAZ toughness. All results at  $\Delta t(800-500) = 3$  seconds. Adapted from Bufalini et al [19].

I don't suppose that many of my readers were aware that manganese and nickel can be so clearly detrimental to HAZ toughness and that carbon may, at least in some circumstances, appear to be beneficial! How often have you come across statements or conclusions in technical papers suggesting that "manganese is detrimental to HAZ toughness" or that "nickel exacerbates the problem of achieving adequate HAZ toughness" and have you ever known the conclusion to be drawn that "carbon may be beneficial to weldability"?

Of course we all know that manganese is an essential 'key' element in steelmaking because of its ability to combine with sulphur and its powerful deoxidation capacity. It is also an inexpensive austenite stabiliser that plays an important role in lowering the  $\gamma/\alpha$  transformation temperature. Equally nickel, alone or in combination with niobium, is well known to be beneficial to CGHAZ toughness in linepipe [20]. So what is wrong with Bufalini et al's results? Why am I presenting them to you? What does it all mean? And, in any event, what has it got to do with niobium?

Well quite apart from the obvious fact that I have been deliberately selective I can assure you that the results portrayed in Figures 4a, b and c are perfectly genuine and, as you will see shortly, are technically explicable when considered in their proper context. However, I can **now admit** that if I had selected from Bufalini et al's extensive data a slower cooling rate or a lower carbon level then both manganese and nickel would be shown to be quite beneficial to CGHAZ toughness. Equally, with ease, their data can be used to show that carbon is more normally very detrimental.

Returning then to the results in Figures 4a, b and c, Bufalini et al [19] were acutely aware of the role of microstructure and fortunately also recorded mean transformation temperatures for all their trial compositions over the full range of simulated HAZ cooling rates studied. Figures 5a, 5b and 5c should be considered in conjunction with Figures 4a, 4b and 4c.



Figure 5. The effect of manganese (C = 0.12%), nickel (C = 0.12%, Mn = 1.5%) and carbon (Mn = 1.0%) on mean transformation temperature. All results at  $\Delta t$  (800-500) = 3 seconds. Adapted from Bufalini et al [19].

It is now clear that the detrimental effects of manganese and nickel at the 0.12 percent carbon level, with a very fast cooling rate,  $\Delta t(800-500) = 3$  seconds, were primarily associated with significant depressions of mean transformation temperature and **in this instance** with poorer martensitic microstructures. Equally, in my **carefully selected** example, the modest beneficial effect of carbon in Figure 4c is explained by the fact that mean transformation temperature in Figure 5c was lowered into a more favourable bainitic range. As already stated manganese and nickel are just as often found to be beneficial and carbon, as we will see later, is far more likely to be detrimental.

Bufalini et al attempted to analyse their extensive data statistically to explore the effects of different alloying elements and transformation temperature variations but it was quickly clear that the composition/microstructural/toughness relationships were too complex to lend

themselves to simple regression analysis. However they did summarise much of their invaluable information in a diagram reproduced here as Figure 6. Their full results, which also contain information on the effects of molybdenum are, even today, well worthy of further detailed analysis.



Mean transformation temperature °C

Figure 6. CGHAZ transition temperature versus mean transformation temperature from Gleeble thermal simulation test pieces. After Bufalini et al [19].

You will appreciate that, at each carbon level and cooling rate, patterns are beginning to emerge.

In a very different alloying regime, Hulka [21] has also produced data that reveals an interesting pattern of effects of manganese and Figure 7 links his observations to microstructural changes. This simply serves to emphasise the point already made that generalised statements about the effect of individual elements should be made with caution.



Figure 7. The effect of manganese on CGHAZ toughness. After Hulka [21].

It was primarily Bufalini's work and other comprehensive research in the same era by Rothwell & Bonomo [22], augmented by my own personal observations, which several years later led me to sketch out Figures 8a and 8b. These conceptual diagrams were first published by Batte & Kirkwood in 1988 [23], and their derivation and rationale is adequately covered in the latter reference.



Figure 8. Influence of carbon content and mean transformation temperature on microstructure and CGHAZ toughness. After Batte & Kirkwood [23].

Figures 8a and b have been cross referenced by various workers to provide explanations for particular results patterns [24, 25] and, in fact, Heisterkamp et al [26] have superimposed data from a range of steel compositions onto Figure 9 to demonstrate the general validity of the proposed model.



Figure 9. Compatibility of a range of results with the model in Figure 8b. After Heisterkamp et al. [26].

This is, however, an appropriate juncture to emphasise that Figure 8b is <u>conceptual</u> and its tentative relationships could never exactly replicate the behaviour of compositions that deviate significantly from those of the steels originally referenced for its derivation. This much is obvious from Figure 9 and, in fact, even more important deviations can be expected particularly when the behavior of more innovative microalloyed steel types is studied.

Nevertheless, for the first time the overwhelming importance of carbon content could be appreciated, as could the reason why no simple regression approach could ever hope to explain the plethora of complex results in the literature. It is now clear that the role of any of the primary alloying elements is to affect transformation temperature and in turn microstructure and toughness. Depending critically on the carbon content and cooling rate then the effect of any particular alloying addition may be observed to be beneficial or detrimental depending on the resulting change in transformation temperature.

Clearly the preferred CGHAZ microstructures are those exhibiting predominantly lower bainite or auto-tempered martensite i.e. of the B1 classification of Bramfitt and Speer [27] in Figure 10.



p = pearlite, m = martensite, a = austensite and c,  $\varepsilon$  = carbide

Figure 10. Classification of transformed microstructures after Bramfitt & Speer [27].

It is also important to appreciate that the B2 and B3 bainite transformations of Bramfitt & Speer's classification rarely proceed to completion in CGHAZ's and by their very nature will result in small pockets of carbon enriched austenite which will duly transform at lower temperatures to lath or twinned martensite (occasionally remaining fully austenitic). Such regions are referred to as MA constituent and in the CGHAZ usually account for no more than 8% of the final microstructure. The mechanism and extent of MA formation is more fully explained elsewhere [28, 29] and for my present purposes it suffices to acknowledge its presence and possible importance.

Ikawa et al [28] have demonstrated that the occurrence of MA is clearly related to cooling rate as shown in Figure 11 and for those who wish to study Ikawa et al's paper in detail it is important to note that their nomenclature for the various bainitic microstructures is more closely aligned to that of Ohmori [30] and is diametrically opposed to that used by Bramfitt and Speers [27]. Ikawa et al's B1 and B3 structures are actually 'B3' and 'B1' in Figure 10 of this paper. That said the observations of the two sets of workers are remarkably consistent and extremely valuable.



Figure 11. Relation between cooling time and fraction of MA constituent in the CGHAZ. After Ikawa et al [28].

It is noteworthy that when  $\Delta t(800-500)$  exceeds 20 seconds there is a very rapid increase in MA and Ikawa et al [28] believe that this corresponds approximately to a transition between <u>their</u> B2 and B1 transformation products. At  $\Delta t(800-500) >$  about 90 seconds, MA is progressively broken down into ferrite/carbide aggregates as much higher temperature transformation products, including polygonal ferrite, begin to appear. Not unexpectedly, increasing carbon content has a significant effect on the fraction of MA encountered and this is well demonstrated by Komizo & Fukado [31] in Figure 12 and supported by Xue et al [32]. Silicon is also similarly implicated [33, 34] and Bonnevie et al [29] suggest that this is because the element is very insoluble in cementite and is therefore concentrated at the interface between cementite and untransformed austenite thus favouring MA formation by stabilizing austenite.



Figure 12. The effect of carbon on the fraction of MA constituent in a CGHAZ. After Komizo & Fukado [31].



Figure 13. Influence of carbon content on the crack initiation temperature of a CGHAZ at a COD level of 0.25 mm. After Komizo & Fukado [31].

Some researchers suggest that it is, in fact, MA which is the dominant controlling factor affecting CGHAZ toughness [32] and Komizo & Fukado [31] attribute the detrimental effect of carbon in Figure 13 to increasing MA constituent. I cannot entirely subscribe to that interpretation because a consideration of Figures 12 and 13, together with my own experience, suggests to me that other factors are at work. In fact whilst MA initially increases with decreasing cooling rate as we move from left to right of Bramfitt & Speers microstructural continuum, in Figure 10, gross microstructure is also changing significantly and I take the view that, **in the CGHAZ**, this is the principal factor controlling toughness. I feel the evidence suggesting that the role of MA is dominant is unconvincing [34]. However its presence is clearly not beneficial to CGHAZ toughness. There is more to be said about MA when we turn our attention to the ICHAZ.

#### Niobium and the Coarse Grained Heat Affected Zone

Niobium is a transition metal found in Group V of the periodic table below vanadium and above tantalum and dubnium. It has a relatively large atomic radius in comparison with iron, a factor that substantially determines its diffusion and precipitation processes in carbon-manganese steels. Consistent with the mystique surrounding niobium it even exhibits an atypical electron configuration in the structure of its electron shells and is the only Group V element to have a single electron in the outermost level. This possibly contributes to the sublety of its behavior and its high affinity for carbon and nitrogen. According to Meyer [35] its unique profile and special effects in steel demonstrate that it is **"more predestined"** to be a versatile microalloying element (MAE) than any other contender. It is certainly the most effective of the MAEs and its specific effects are so powerful that certain desired properties in steel can be obtained with concentrations as low as 1 niobium atom in 10,000 atoms of iron [35].

In respect of the CGHAZ the effect of dissolved niobium in retarding the  $\gamma/\alpha$  transformation is well established and widely reported. Poole et al [14], for example, have clearly demonstrated the magnitude of the effect in a C-Mn- Mo-Ti- Nb (0.034%), X80 pipeline steel, Figure 14.



Figure 14. The effect of niobium on austenite decomposition kinetics and transformation temperature. After Poole et al. [14].

However, there is still uncertainty regarding the precise mechanism by which niobium exerts its remarkably strong influence [36, 37] though there is considerable agreement that, when sufficient niobium is dissolved in the gamma matrix,  $Ar_3$  is lowered on cooling by about 10 °C per 0.01 percent dissolved niobium [38, 39]. It is certainly not my intention to enter into the debate in depth but my own observations and my understanding of the relevant literature lead me to question the 'solute drag' model [36] and favour the explanation tentatively forwarded by Fossaert et al [37]. The latter author's observations support earlier work by Thomas & Michal [40] suggesting that niobium atoms in solid solution may well segregate preferentially to austenite grain boundaries where they affect the nucleation of the austenite transformation products. This is feasible because the niobium atoms have a large misfit with the iron matrix, and austenite grain boundaries may become favourable sites for them to diffuse to [41].

The precise mechanism whereby the kinetics of transformation are subsequently affected remains to be clarified but Fossaert et al [37] suggest that the effect appears to be analogous to that of boron in steel and is found to be largely independent of austenite grain size. This idea is supported by Furuhara et al [42] who, following detailed studies on the 'incomplete' bainite transformation in low carbon niobium steels, concluded that niobium does not inhibit ferrite growth <u>but</u> ferrite nucleation. Further support for this interpretation can be derived from recent work by Li et al [43] who cleverly studied the retardation effect, on isothermal transformation, of niobium in 0.1% carbon, 1% manganese steels at constant austenite grain size. Li et al concluded that niobium must be segregating to austenite grain boundaries and that its effect on the transformation from austenite to ferrite is stronger in the case of reconstructive transformation than in lower temperature displacive, bainitic or martensitic, transformations.

Reporting on their studies on a 0.15 percent carbon, 1.5 percent manganese, 0.035 percent niobium steel Fossaert et al's key diagrams, reproduced here as Figures 15 & 16, support their hypothesis.



Figures 15 & 16. The effects of austenite grain size and niobium in solution on the critical cooling rate required to produce 95 percent martensite. After Fossaert et al. [37].

The critical cooling rate necessary to produce 95 percent martensite clearly decreases markedly once austenite grain size increases and also as more niobium is dissolved in austenite. However once sufficient niobium is in solution then further increase in grain size has very little effect on the critical cooling rate.

Pursuing this hypothesis further, rapid HAZ thermal cycles which fail to allow much niobium to re-dissolve in austenite would result in existing Nb(CN) precipitates exerting a significant grain refining effect as in Figure 17 which might well have the effect of decreasing hardenability whilst slower cycles which permit the dissolution of the precipitates should then maximise the expected effect of niobium on hardenability. In the latter context the role of carbon content will obviously be very significant; this is clear from Figure 18. This is a simplistic analysis because Nb(CN) precipitates are, in some circumstances, also capable of retarding ferrite grain **growth** during transformation.



Figure 17. The effects of niobium and thermal cycle on CGHAZ austenite grain size. After Hannerz [44] but modified using data from other sources.



Figure 18. The effect of carbon, nitrogen and austenite temperature on niobium in solution. After Klinkenberg [45].

In very slow thermal cycles, with a longer dwell time in the austenitic region and slower cooling rates for example with  $\Delta t(800-500) > 200$  seconds, then the possibility of re-precipitation of Nb(CN) during cooling must be considered.

# So how does all this theory stand up in practice?

There are endless publications in the literature but, for the purposes of this paper and the rationale that I am attempting to develop, I will rely on a relatively small number of relevant and representative contributions. I will first, once again, ask you to consider important data from the 1970's from papers which were regarded as significant at that time and will then compare and contrast the findings from that work with results and conclusions from selected more recent publications.

In the late 1970s Rothwell et al [46] carried out an interesting and fairly comprehensive study on the role of niobium in steel compositions potentially suitable for various linepipe grades. The results that I particularly want to focus on are derived from 12mm plates with nominal carbon and manganese levels of 0.09 percent and 1.3 percent respectively. Niobium was incorporated into the study at four levels: zero percent, 0.034 percent, 0.09 percent and 0.14 percent. Submerged Arc Welds were made at heat inputs ranging from 1.4 to 7.5 kJ/mm corresponding approximately to  $\Delta t(800-500)$  from 16 seconds to 160 seconds. The completed welds were notched into the CGHAZ and subjected to instrumented Charpy impact testing carried out over a range of temperatures to determine yield load fracture transition temperatures. In parallel, samples of the steels were studied using a high-speed dilatometer to generate Continuous Cooling Transformation diagrams. There are many other studies from this era which report similar trends in respect of the influence of niobium on CGHAZ toughness but the results of Rothwell et al's study are uniquely valuable because they allow the toughness results obtained to be considered in the context of the CCT diagrams and the microstructures generated.

Figure 19 presents their toughness data and it is immediately apparent that only at higher heat inputs with  $\Delta t(800-500) > 80$  seconds and at the higher microalloying levels does any systematic adverse effect of niobium become apparent. It is difficult to be precise but for heat inputs below about 3 kJ/mm { $\Delta t(800-500)$  below 70 seconds} it would appear likely that niobium, at all levels in the carbon and manganese regime studied by Rothwell et al [46], should be beneficial or neutral to CGHAZ toughness. Bearing in mind that 3 kJ/mm in a 12 mm plate produces a relatively slow cooling rate  $\Delta t(800-500) = 70$  seconds, and that this cooling rate also equates to approximately 4.5 kJ/mm in 20 mm plate or, in the 3D heat flow regime (for anything above about 25 mm) a heat input of around 7.5 kJ/mm the conclusion concerning the influence of niobium should apply over an extremely wide range of practical welding processes.



Figure 19. The effects of niobium and heat input on yield load fracture transition temperature of the CGHAZ. After Rothwell et al [46].



Figure 20. The effects of niobium and cooling rate on the CCT ferrite start temperature of the CGHAZ. After Rothwell et al [46].

To explain these benefits of niobium the CCT diagrams and associated microstructural observations become invaluable and Figure 20, adapted from Rothwell et al [46] focuses on the effects of niobium and cooling time on ferrite start temperature. Note that the cooling times in Figure 20 do not equate directly to those associated with the heat inputs in Figure 19. As can be appreciated from Figure 20 the fastest thermal cycle reveals no effect of niobium, at any level, on 'start' temperature. Even at  $\Delta t(800-500) = 12$  seconds the lowest addition of niobium appears to have no effect. However, at the 0.09 percent niobium level and above, the latter cooling rate produces a depression of 'start' temperature of > 25 °C. According to the CCT data a cooling rate equivalent to  $\Delta t(800-500) = 30$  seconds, just a bit faster than that of the 2.4 kJ/mm weld, and additions of niobium at the 0.035 percent and 0.09 percent level depress the ferrite start by at least 50 °C. At this intermediate cooling rate the highest niobium level produces what seems to be an anomalous result and no depression of 'start' temperature. There then appears to be a reversal in behavior, and the very slowest cooling rate of the CCT data clearly suggests that the higher levels of niobium are, once again, causing a very significant depression in start temperature.

These observations are broadly consistent with the model outlined previously on the basis that the fastest thermal cycles only enable sufficient niobium to dissolve in austenite when a high level of the element is present and then only at  $\Delta t(800-500) > 12$  seconds. The modest improvements in HAZ toughness noted at low heat inputs with niobium appear, from Rothwell et al's metallography, to be associated with austenite grain refinement but, in the absence of measurements, this conclusion is somewhat subjective. At slower heating and cooling rates, although there are anomalies, there is clear evidence of the predicted depression of transformation start temperature that must be the result of a significant level of soluble niobium in the austenite. Rothwell et al speculate that some of the anomalous observations may be explained by the fact that with the very slowest cycle the time spent above 1000 °C is about 390 seconds which is sufficient to ensure that most of the niobium will be in solution and that there will be significant austenite grain growth (Both these factors contributing to increased hardenability and acting to prevent excessive polygonal ferrite formation which might otherwise have been expected at such slow cooling rates).

Rothwell et al.'s microstructural observations are also consistent with the general theory outlined earlier. At the fastest cooling rates, irrespective of niobium level, the transformed HAZ microstructures consist of mixtures of martensite and bainite with parallel ferrite laths and a very small percentage of MA constituent. As cooling rate decreases, in the absence of niobium, grain boundary ferrite is increasingly observed and coarser bainite exhibiting randomly orientated ferrite laths replaces the previously described bainite. Eventually, MA also decreases above a certain heat input consistent with Ikawa et al's observations [28]. Slower cooling rates produce little microstructural change at the lowest niobium level but, at higher levels, grain boundary ferrite is progressively replaced by lower temperature transformation products leading to improved toughness. As might be expected from Figure 20 there are anomalies at certain cooling rates so the competing effects of niobium in solution, grain growth and cooling rate come into play.

In another important 1970s contribution, Dolby [47] reported results from a study on 25 mm ship plate with nominal carbon and manganese levels of 0.16 percent and 1.25 percent respectively. Unfortunately there were certain compositional variations which require careful consideration when drawing conclusions since the 'base' carbon-manganese as rolled plate had a very high

sulphur level (0.046%) and a very low silicon level (0.03%) whilst the comparison, normalized, plates were carbon-manganese (Al + Si killed), carbon manganese (0.019% Nb; Al + Si killed) and carbon-manganese-0.056 percent niobium, silicon killed with a very low aluminium content (0.011%).

Nevertheless, the results from Dolby's submerged arc (twin wire) and Electro Slag (ES) welds are important and add significantly to our understanding of the role of niobium in the HAZ.

Dolby's welds were primarily assessed using COD test pieces notched into the CGHAZ but additional Charpy results were also recorded in the case of the ES welds. The submerged arc welds were made at heat inputs in the range 5-7 kJ/mm producing thermal cycles with  $\Delta t(800-500)$  in the range 60 to 111 seconds and the ES welds at 25 kJ/mm providing a measured  $\Delta t(800-500)$  of 200 seconds. Figure 21 below shows selected HAZ, COD results at -40 °C.



Figure 21. The effect of heat input on critical COD values for CGHAZs in 25 mm ship plate. After Dolby [47].

It should be noted that the much lower yield strength carbon-manganese 'base plate' exhibited consistent COD HAZ values > 0.5 mm. For the reasons already stated, care must be taken in drawing definitive conclusions from Dolby's study but it seems safe to conclude that niobium treated steels with **higher carbon levels**, which were common at the time, were not well suited to maximising HAZ toughness at higher heat inputs (at least as measured by the COD test).

Fortunately, Dolby [47] made a number of interesting microstructural observations in his paper. In addition to noting a progressive increase in austenite grain size (prior to transformation) as heat input increased (the C-Mn steel exhibiting by far the highest values), it was noted that the HAZ austenite grain boundaries of the carbon-manganese and carbon-manganese-aluminium submerged arc and electro slag welds were delineated with proeutectoid ferrite the remainder of the microstructures consisting of bainite in various morphologies. In the HAZs of the niobium steels pro-eutectoid ferrite was absent except in the electro slag welds and the dominant microstructural constituent was upper bainitic in character with colonies of parallel ferrite and carbide laths reminiscent of Rothwell's intermediate cooling rate products [46]. Small pools of up to 3% MA, considered by Dolby to be primarily retained austenite, were noted in the submerged arc welds (except in the C-Mn steel) but were notably absent in the ES welds.

Interestingly, Dolby found no direct evidence of Nb(CN) precipitation even in the most slowly cooled Electro Slag HAZs.

Dolby concludes from his work that niobium is clearly depressing transformation temperature but that the overall effect of the element (at this C and Mn level) will depend on the net result of a number of opposing interactions. Dolby attempts to summarise these in a vector style diagram, Figure 22, following the presentation format employed by Garland & Kirkwood [48] for acicular ferrite weld metals some years earlier.



Figure 22. Schematic diagram showing various possible effects of niobium on CGHAZ toughness of high heat input welds. After Dolby [47].

Dolby's results are also entirely consistent with the model described previously and underline one of the conclusions of the 1980 review [10] that carbon is particularly detrimental to HAZ toughness in niobium containing steel HAZ's when higher heat input welding processes are employed.

Turning now to much more recent information Zhang et al [49] contribute further to our understanding of the role of niobium in more complex microalloyed steels. Studying simulated CGHAZ microstructures with Charpy test specimens machined from 21 mm plates of quenched and tempered storage tank steels and simulated welding thermal cycles 3-12 kJ/mm providing  $\Delta t(800-500)$  times in the range 11 to 125 seconds Zhang et al explored the role of niobium in 0.07 percent carbon, 1.36 percent manganese steels (containing Ni+Cr+Mo = 0.37%) and **importantly** 0.04 percent vanadium and 0.01 percent titanium. Their results in Figures 23 & 24 confirm the well noted effects of niobium in depressing transformation start temperature over a wide range of cooling rates and suggest that lower temperature, harder, microstructural constituents associated with lower temperature transformation products lead to reduced Charpy toughness at -15 °C.



Figure 23. The effect of niobium (0.026%) and heat input on transformation start temperature and Figure 24. The effect of niobium (0.026%) and heat input on CGHAZ toughness. After Zhang et al [49].

Signes & Baker [50] and Rothwell [46] have both previously demonstrated that the role of Nb is extremely complex in the presence of vanadium and a full treatment of this interaction is out with the scope of this paper. However, at higher heat inputs with both microalloying elements present there can be an enhanced depression of transformation temperature over a wider range of cooling rates and Zhang et al's microstructural observations are consistent with those of the workers referred to earlier. At low heat inputs with fast thermal cycles, where Nb(CN) precipitates do not completely dissolve, fine grained autotempered martensite results which, although harder than the equivalent niobium free transformation product is still extremely tough. As heat input increases and the full effect of niobium in solution comes into play then, <u>in this particular alloying system</u>, the net effect of the presence of niobium is clearly negative and coarse granular bainite progressively replaces the more favourable structures. For those interested to pursue the matter further, Zhang et al's paper contains a wealth of valuable transformation of which reveals remarkable compatability with the work reported previously.

Finally I wish to draw your attention to a paper which I consider to be of particular significance. In 1998 Hattingh & Pienaar [25] studied a range of steel compositions potentially suitable for use in > 50 mm thickness normalized plates for LPG storage tank applications. Their work was instigated to study the validity of steel specification niobium limits stipulated by certain construction companies in circumstances where the steelmakers would have preferred more flexibility to exceed such arbitrary limits to obtain the required levels of plate strength and toughness more readily. For the purposes of their study forged billets of aluminium treated steels with nominal manganese levels of 1.2 percent were rolled down to 11 mm thickness using a procedure designed to simulate the rolling of plates with a final thickness of 50 mm. Using a fairly robust experimental design Hattingh & Pienaar [25] then systematically studied the effects of carbon and niobium on CGHAZ toughness using Gleeble thermal cycles designed to represent the welding of 50 mm plates at heat inputs of 1.5, 3.0 and 6 kJ/mm with a peak temperature of

1350 °C in each instance. Reference to the 3D line of Figure 2 provides estimates of  $\Delta t(800-500)$  of 7, 18 and 34 seconds respectively for the three heat inputs studied. From their comparison with practical welds, the authors suggest that the thermal cycles used were representative of the CGHAZ at a position 0.4 mm from the fusion line. The following three diagrams are selected and reproduced directly from their report as Figure 25 (a, b & c). I should note that the authors stress that the absolute levels of toughness portrayed are comparative and not necessarily what would be experienced in the very narrow zones of real welds.



Figure 25. The effects of carbon, niobium and heat input on CGHAZ toughness (50% FATT). After Hattingh & Pienaar [25]

These three dimensional presentations are particularly useful and clear patterns of behavior can be identified. Perhaps the most important observation, which comes as no surprise, is that carbon is 'bad news' and that levels above about 0.14 percent are particularly unwelcome irrespective of

niobium level. High carbon in conjunction with high niobium is clearly detrimental at the highest heat inputs as we have seen earlier in this paper. Interestingly the best results at heat inputs up to 3 kJ/mm appear to support the general trends of Figure 8b and occur at around the 0.12 percent carbon level with niobium levels of up to 0.065 percent. This also supports the findings of the original 1980 review [10]. At 6 kJ/mm the optimum results are recorded with carbon levels below 0.08 percent and seem to be relatively insensitive to increasing niobium up to 0.065 percent. Hattingh & Piennar's [25] 'base' compositions, in terms of manganese, silicon and aluminium are not greatly different from those of Rothwell [46] and it is unfortunate that the former authors did not record specific transformation temperature data. Nonetheless, while Hattingh and Piennar's optimum results were recorded at slightly higher carbon levels than studied by Rothwell there is a considerable degree of consistency in the nature of the microstructural observations made.

I will not dwell on Hattingh & Piennar's [25] detailed interpretation of their results and it suffices to observe that higher carbon levels led to hard untempered lower bainite and martensite at the fastest cooling rates and coarse bainite with lath type structures as cooling rate decreased. In the presence of niobium and high carbon the authors suggest that precipitation of Nb(CN) further embrittles already poor microstructures particularly at the highest heat input where excessive grain growth and coarse microstructures were observed. Optimum toughness in all cases was associated with combinations of autotempered martensite and fine bainite. The critical role of transformation temperature is, yet again, clearly demonstrated.

We could readily continue to look at many other contributions to the literature but, I believe, the data already considered presents us with a sufficiently clear picture. Like many of the primary alloying elements, manganese, molybdenum, chromium and nickel, niobium acts to depress the temperature at which austenite transforms to lower temperature products; see Table I. However, there the similarity ends because niobium can be used in such low amounts to provide extremely powerful effects thus avoiding any danger of over alloying and excessive solid solution hardening. Additionally it has the added flexibility of acting to refine austenite grain size in the CGHAZ particularly at lower heat inputs and this provides steel and welding metallurgists with a further opportunity to tailor compositions to match particular end products and manufacturing routes. The precise effects of niobium, relative to those of the common primary alloying elements, will clearly vary with overall composition and welding thermal cycle but the following table, which I have derived from many of the papers referred to earlier provides a powerful illustration of the potency of niobium with intermediate thermal cycles in the range  $\Delta t$  (800-500) = 40-50 seconds.

Element	Depression of mean	Observations	
	transformation temperature		
carbon	7.5 °C / 0.01%	Valid from .06 to .12 % C	
manganese	10 °C / 0.1%	Valid from 1 to 2% Mn	
nickel	5.6 °C / 0.1%	Valid from 0 to 0.75% Ni	
molybdenum	No consistent effect,	Valid from 0 to 0.7% Mo	
-	usually small +ve or -ve		
niobium	10 °C / 0.01%	Valid for Nb in solution up to 0.06%	

Table I. Transformation Temperature Depression Rates of Selected Elements Derived from 0.06 % C, 1.5% Mn steels [  $\Delta t$  (800-500) = 40-50 secs]

A full understanding of the complex role of niobium and its various precipitates will continue to elude us for some time but we have surely learned enough to be able to predict how to judiciously apply the element to its best advantage. Carbon is particularly unwelcome in most welding situations and, with or without niobium, its presence should clearly be restricted as much as practicable if the best CGHAZ properties are to be achieved. However, because of the high affinity which carbon and niobium have for each other and the various interactions which can result, the evidence suggests that niobium specifically will be much more effectively utilised over a wider range of practical welding heat inputs when carbon levels are carefully controlled. If niobium bearing steels are to be effectively used at very high heat inputs with processes such as electro slag welding then carbon levels must be well below 0.1 percent and extra primary alloying will be required to maintain strength. Austenite grain refining can be recovered using carefully controlled Al/N ratios [51] and or titanium treatment [52]. The success of any grain refining effort will dictate the selection and amount of primary alloying required. Considerable evidence suggests that combinations of niobium and vanadium are unhelpful in steels for the highest heat input welding processes.

Before describing the most significant developments of recent years I need to say a few words about the ICHAZ

# The Intercritical Reheated Coarse Grained Heat Affected Zone (ICHAZ) and the Role of Primary and Microalloying Elements

You will be relieved to learn that this section of my contribution will be significantly shorter even though ICHAZ's tend to be more extensive than their adjacent CGHAZ's. This is a natural consequence of the slower cooling rate that usually results from the second or subsequent thermal cycles. I have already dealt with all the basic metallurgical concepts required to discuss this zone.

I referred earlier to the presence of MA constituent in the CGHAZ which is usually very high in carbon content as a consequence of its mechanism of formation and partitioning. Concentrations of carbon around the 1 percent level are not uncommon in the small regions of MA in the original CGHAZ [53] and it is these regions which, therefore, re-austenitise first in the ICHAZ. These MA regions then grow in extent and absorb more carbon from the surrounding matrix and by the time the whole zone is cooling again they can easily account for around 15 percent of the transformed microstructure in mid range heat input welds. Small MA regions tend to exhibit a carbon gradient with the highest carbon levels at their periphery; this means it is possible to find martensitic cores and austenitic envelopes. Analysing simulated CGHAZ and ICHAZ regions in 0.08 percent carbon, 1.55 percent manganese, 0.3 percent nickel, titanium treated steels, Bonnevie et al [29] showed that, a cooling rate of  $\Delta t(800-500) = 30$  seconds, from an initial max temp of 1350 °C, followed by an 810 °C second pass, produced a significant increase in the mean size of the MA population from 1.4 µm to 2.0 µm. This resulted because the volume fraction of the MA > 2  $\mu$ m had increased from 20 to 36 percent, of the total MA present, and the volume fraction of the MA > 3  $\mu$ m had increased from 5 to 15 percent. Obviously a low heat input second pass with a fast thermal cycle would minimise the growth of these MA regions. These important observations of Bonnevie et al. [29] are summarized in the following table adapted from their paper.

		Total MA population	MA with L max > 2 μm	MA with L max > 3 μm
CGHAZ (1350 °C)	Mean size	1.4 μm	2.8 µm	3.9 µm
	Proportion of total population	100%	20%	5%
ICHAZ (1350 °C+ 850 °C)	Mean size	2.0 µm	3.2 µm	4.3 μm
	Proportion of total population	100%	36%	15%

Table II. The Effect of a Second Thermal Cycle on MA Size and Distribution,  $\Delta t$  (800-500) =30 s. After Bonnevie et al [29]

As observed previously, when discussing the CGHAZ, the incomplete bainitic reaction inevitably results in the formation of MA and the amount present in the CGHAZ is clearly the dominating factor in controlling how much will be observed in the ICHAZ. However Matsuda et al [34] believe that certain elements such as carbon and silicon are particularly influential. Carbide forming elements such as chromium, molybdenum, niobium and vanadium, it is suggested [34], also encourage the retention of MA by increasing the hardenability of small reaustenitised areas during the second thermal cycle. However the effect of any element whether primary or microalloying will depend upon the transformation regime which results from the specific alloying addition.

There does, however, appear to be a particularly strong influence of silicon in the ICHAZ and Bonnevie et al [29] have demonstrated how the element appears to increase the density of the MA particles which they believe are most detrimental to ICHAZ toughness. Figure 26 shows the effect of silicon and how the peak temperature of the second pass is of considerable importance in influencing the density of MA produced.



Second peak temperature (°C)

Figure 26. The effect of silicon and second pass peak temperature on the occurrence of MA constituent (Numbers of larger constituents/mm<sup>2</sup>). After Bonnevie et al [29].

Some researchers have suggested that niobium steels encourage the retention of higher levels of MA in the ICHAZ [39, 42] but a close study of their actual observations leads me to believe that it is the transformation temperature and the nature of the transformation product that largely dictate the amount of MA present. It is obvious that, if a comparison is made between a carbon-manganese 'base' steel which at a given cooling rate produces a significant fraction of polygonal ferrite and a niobium treated steel, more MA will be observed in the HAZ of the latter. However, this is not because niobium promotes MA but because a slightly lower temperature intermediate bainitic microstructure naturally exhibits increased MA as previously discussed. There is, however, the possibility that niobium as a carbide-forming element with a powerful affinity for carbon (which concentrates in MA) could result in some effect of the nature suggested [39]. Nevertheless it is surely obvious that the principal factor affecting the amount of MA in the ICHAZ is the amount afready present in the CGHAZ. This is in turn determined by gross microstructure as previously explained.

A much more intriguing possibility is that MA formed in niobium bearing steels may preferentially absorb much more of the element than might be expected. This is not just as a result of the powerful affinity that carbon and niobium have for each other or normal transformation partitioning but because, at the carbon levels present in MA (around 1%), Palmiere et al [54] have demonstrated that austenite has an enhanced capacity to retain much more niobium in solution. The explanation for this phenomenon, which seems inconsistent with the trend in Figure 18, is well beyond the scope of this paper. However, this concept, in theory, might suggest that, while the amount of MA may not be directly affected by niobium, the hardenability of the constituent could well be. Depending on the overall alloy content and cooling rate it is conceivable that carbon and niobium enriched MA could transform at much lower temperatures and may even be fully retained as austenite in some instances. The net result

of such an effect, on toughness, need not be detrimental but there is a further story here for another occasion!

It seems probable, though very difficult to quantify, that the increased volumes of larger MA regions in the ICHAZ may be the important factor controlling toughness and may **in some circumstances** be a significantly more important factor **than in the CGHAZ** [55]. Thus since its presence cannot be entirely avoided except in the lowest heat input welds it would seem to be prudent to take obvious steps to reduce the problem. The evidence that I have reviewed in this paper would strongly suggest that carbon and silicon levels should be reduced to the lowest levels practicable commensurate with acceptable alloy design.

# The Way Forward

If you have followed the gist of my deliberations to date, you will not be too surprised when I suggest that there is no single way forward. Instead there are many options when you are seeking to optimise the composition of a niobium bearing structural or linepipe steel to meet any particular combination of parent plate strength/toughness and specified HAZ toughness. However there has been an important and innovative breakthrough in recent years for higher strength linepipe of X80 strength and above which also expands our knowledge of the remarkable possibilities that niobium provides as a consequence of its unique characteristics.

Ultra low carbon steels employing the full hardenability effects of much higher niobium levels in solution were first investigated and quite thoroughly researched decades ago, in the UK by Dewsnap & Frost [56] and in US Steel by Gray et al [57], but much of the knowledge gained at that time was set aside and, to a degree forgotten, until the concept was gradually revived by forward thinking metallurgists, more progressive steelmakers and committed pipe fabricators prepared to embrace the possibilities of a radical new approach.

This thinking, over a period of years, has resulted in the evolution of a class of linepipe steels specifically designed to take maximum advantage of niobium's special effects when employed with ultra low carbon contents circa 0.03 percent. Such steels which typically contain about 0.1 percent niobium and 1.7 percent manganese are further alloyed with elements such as copper, chromium, nickel and molybdenum to produce compositions which allow thermomechanical processing at higher temperatures than normal. Steels of this class also harness the ability of titanium in stoichiometric proportion with nitrogen to provide additional retardation of grain growth during heating. These steels are known as "High Temperature Processed" (HTP) steels and the flexibility of the approach is best demonstrated with reference to the plethora of alloy routes that have been successfully explored by individual companies [4, 58, 59,] in practice to achieve the X80 linepipe grade.

Vast tonnages of such steels have already been successfully incorporated into completed large diameter HSLA linepipe projects in strengths up to X80 and wall thicknesses up to 30 mm. By far the most dramatic of these is the 2<sup>nd</sup> West-East Chinese Gas pipeline project. Employing X80 pipe at 18.4 mm wall thickness, the latter includes some 2.75 million tonnes of material [4], a remarkable achievement and a massive vote of confidence for this relatively new technology.

From the welding standpoint, the most exciting thing about these steels is their ability to be welded over an extremely wide range of heat inputs to provide a consistent fine-grained bainitic microstructure with excellent CGHAZ toughness. Because of the very low carbon content more

niobium is taken into austenitic solution during welding, Figure 18, and this allows the maximum effect on ferrite retardation to be obtained over a wide range of heat inputs and cooling rates. A typical CCT diagram is included below, Figure 27, which indicates how the ferrite 'nose' has been displaced so far to the right that a bainitic transformation product is readily produced, as already indicated.



Figure 27. CCT diagram for simulated CGHAZs with a peak temperature of 1350 °C of an HTP steel with 0.03 percent carbon, 1.75 percent manganese and 0.1 percent niobium. After Hulka [21].

This 'nose' displacement precisely mirrors the effect produced by boron at low carbon levels and, in my mind, lends great weight to the tentative explanation of the mechanism of the niobium 'hardenability' effect forwarded by Fossaert et al. [37] as discussed earlier in this paper.

With such reduced carbon content, the toughness of the resulting bainite is excellent over a wide range of cooling rates from 3 °C/sec to > 100 °C/sec encompassing most of the common welding processes. Additionally the occurrence of MA in the CGHAZ is rare. This factor, together with low silicon contents (usually < 0.2%), ensures that MA does not become a factor of concern even in the ICHAZ. Figure 28 compares the CGHAZ toughness obtained from a representative X80 HTP steel with that recorded from a more normal higher carbon, Nb-V X70 composition.



Figure 28. Comparison of simulated CGHAZ toughness results from HTP steel with those from a conventional X70 grade. After Hulka[21].

Stepanov et al. [60] have also reported similar results for a composition of this class and have demonstrated that their particular variant of HTP steel is suitable for all welding processes likely to be encountered in pipe fabrication (including site work) over a cooling range from 5 °C/sec to 120 °C/sec. Even at the slowest cooling rates CV values above 100 J/cm<sup>2</sup> at -40 °C were obtained.

There is clearly scope to explore this low carbon high niobium philosophy for other potential, thicker plate, applications in the structural and offshore fields even where the HTP concept is irrelevant and normalising is a much more likely supply route. There is no reason to suppose that the resulting HAZ behavior will not follow a similar pattern even though Manohar et al [61] have highlighted the potential significance of the applied thermo-mechanical deformation processes and their influence on the CCT behavior of niobium bearing steels. As discussed later the role of stress relieving where appropriate would, of course, also have to be considered.

#### Heat Affected Zone Toughness in Perspective

In most systematic studies of compositional factors affecting HAZ toughness a base carbonmanganese composition is usually chosen to provide an indication of the metallurgical effects of the addition of one or more primary (or microalloying) elements. It should always be remembered that this 'base' is highly unlikely to be capable of delivering the combination of strength and toughness of interest to the researcher and it is the properties of the various compositional options which **actually deliver** acceptable properties which should be focused on rather than, as is often the case, the change in properties which results from the addition of the element being studied without any consideration of the differences in yield strength.

# So how tough is tough enough?

There is no doubt that in plate thicknesses > about 30 mm the risk of brittle fracture is still a serious consideration and correlations between COD tests and wide plate tests are often valuable to define acceptable standards for all regions of welded joints. Heisterkamp et al [26] have described an approximate 'envelope' of carbon and niobium levels in which, on the basis of collated data, "good" COD results at -10 °C can be readily achieved in thicker steels. Of course whether "good" is good enough depends on the completion of an appropriate Engineering Critical Assessment (ECA) for any particular application. However, I have reproduced their diagram below without all the individual data points and have taken the liberty of speculatively modifying it to admit the success achieved with ultra low carbon, 0.1 percent niobium, HTP steels.



#### Niobium content %



This diagram is not intended to infer that adequate levels of toughness can only be achieved within the 'envelope' delineated in Figure 29 and it is, strictly speaking, only relevant to the

steels from which it was derived. However, it reiterates the importance of the carbon–niobium interaction and provides a degree of guidance for structural steel compositional design.

In this regime individual company specifications will continue to dominate and fitness for purpose assessment will dictate acceptable fracture toughness levels.

Turning to thinner plates, particularly in linepipe applications, there have been a number of interesting reviews/assessments of how best to measure the toughness of both seam and girth weld HAZs [62, 63]. There are so many options in respect of CV versus COD, specimen orientation, notch type and acuity, etc. that, it is almost inevitable, if you test any welded joint often enough in numerous ways you will eventually identify a small, often microscopic, zone of lower toughness. I guess every joint has its Achilles heel but does it really matter?

It is certainly the case in pre 1970's pipes welded with outmoded techniques that there are still latent problems waiting to surface [64] and indeed there have been spectacular failures of old 1940's girth welds from pre existing weld defects caused by excessive stress applied externally in more recent years [65]. However more contemporary failure statistics from pipes fabricated since about 1990 [66, 67] suggest that improved materials, modern fabrication techniques and vastly improved inspection techniques have combined to virtually eliminate the welded zones of HSLA pipelines from being an area of serious concern. Failures in modern-day pipelines are extremely rare, and the most common causes are external interference and inadequate corrosion protection.

It is considered that the general level of pipe material and welded zone toughness is now such that the risk of brittle fracture can usually be set aside at least for pipes up to 25 mm in thickness [68] and as long as excessive texturing which might, in extremis, allow a form of ductile fracture propagation to occur, is avoided [69] then the required toughness for welded joints can be set on the basis of plastic collapse criteria [68]. Returning to my mythological theme and the 'Achilles Heel' analogy, even Achilles would not have perished if he had been wearing more appropriate footwear instead of the leather sandals he is usually portrayed in! Modern welded joints do, however, usually have a form of protection; it's called overmatching and it has been shown that such protection combined with the factors already discussed above reduces the acceptable level of flawed girth weld zone toughness to quite modest values when plastic collapse is the potential failure mode under consideration.

Using correlations between curved wide plate tests, incorporating 3 mm deep surface breaking root cracks, and Charpy testing, the European Pipe Research Group (EPRG) has established that, for pipe wall thicknesses up to 25.4 mm and for strengths up to X70, overmatched welded zones only require an average CV of 40 Joules (minimum individual result 30 Joules) at the minimum operating temperature of the pipeline [70]. For wall thicknesses < 12.7 mm the figures can be reduced to 27 Joules average and 20 Joules as an individual minimum.

These are readily achievable values with most modern HSLA approaches to steelmaking and fabrication and, while we should not be complacent about the toughness of any region of modern day welded joints in linepipe, we should equally accept that it may be unnecessary to produce excessively demanding specifications for weld metals and HAZs which place unreasonable constraints on suppliers and welding engineers.

In this paper, I have quite deliberately confined my attention to the as-welded situation since a full consideration of the effects of post weld heat treatment would have extended this

contribution beyond what I considered to be reasonable. Also modern low carbon plate steels, even in thicknesses up to 100 mm and beyond, rarely require to be subjected to post weld heat treatment. However, there are a few general observations that it is pertinent to make at this juncture.

When niobium bearing steels and weldments are stress relieved there are numerous effects to consider. In many cases the tempering and effective removal of MA constituent from HAZ microstructures offsets any potentially detrimental effects of precipitation hardening but this will not always be the case. Fortunately, post weld heat treatment is usually carried out with the intention of reducing residual stresses rather than effecting any specific metallurgical changes and in this context it should always be remembered that the successful elimination of residual stresses significantly increases the tolerance of the welded joints to the presence of defects even if the fracture toughness is moderately reduced by the procedure.

# **Concluding Remarks**

I hope I have been able to demonstrate how it is all too easy to leap to erroneous conclusions about the role of any specific element in the context of effects on HAZ toughness as has clearly happened with niobium from time to time. This is frequently the result of ineffective experimental design or a 'blinkered' view of available information and I would urge the current generation of researchers to avoid these pitfalls and to approach their research with an open mind and without a preconceived notion of what it is they expect to establish! During my reading, whilst preparing this paper, I came across several well meaning contributions to the literature in which the conclusions drawn by the authors did not entirely accurately reflect their presented data. In a few cases the claimed effect of a particular element could not be substantiated because a closer examination revealed that the experimental design was fundamentally flawed and that potentially significant compositional variations or prior thermal history variations between the steels being examined had not been highlighted. The behavior of different steels must be compared on the basis of a common denominator.

The foundation of accurate interpretation lies in experimental design and when this is adequately addressed and the study is beyond reproach then there is much less likelihood of misleading conclusions being drawn.

In sixteenth century England, when 'wit' was the general term for intellectual powers, Sir John Davies, an important writer and lawyer, managed to express my concerns much more poetically when he conjured up the following rather astute philosophical observation.

"But if a phrenzy do possess the brain, It so disturbs and blots the form of things, As fantasy proves altogether vain, And to the wit no true relation brings. Then doth the wit, admitting all for true, Build fond conclusions on those idle grounds;"

Having addressed your experimental design then think carefully about how your results will be received.

Remember your meticulous research is useless unless it can be explained to and be understood by the people with the power or influence to make policy leading to the evolution of progressive material and welding specifications.

Do not be afraid to embrace the challenge of the rapidly advancing technology especially in the field of HSLA steel developments. You have seen how readily apparent hurdles can be overcome and no longer have to fear the mythologies of earlier literature. Indeed, through recent developments, steelmakers and welding metallurgists have been afforded tantalising glimpses of the full potential of higher levels of niobium and how this element will, inevitably, feature for the foreseeable future in modern HSLA steel design.

Finally, I came across two old proverbs which those who are determined to succeed would do well to heed. Though different in their derivation they have similar connotations.

From China: "Be not afraid of growing slowly; be afraid only of standing still"

and

From Brazil: "One who does not look ahead remains behind"

# Addendum

In Classical Mythology Tantalus and Niobe, as mere mortals, eventually provoked the anger of the Gods and were severely punished with Niobe acquiring the tag of 'Goddess of tears'. Now, however, after the passage of the millennia and in the light of a unique contribution to technological development, it is perhaps time to accord to Niobe and her namesake niobium a more deserving place in a new chapter of present day Mythology. In recognition of the importance of this influential gathering to HSLA steel development I propose that we should confer on Niobe the title of Goddess of Araxá.

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