

DEVELOPMENTS IN NIOBIUM STEELS FOR LINEPIPE APPLICATIONS



COMPANHIA BRASILEIRA DE
METALURGIA E MINERAÇÃO



**COMPANHIA BRASILEIRA DE
METALURGIA E MINERAÇÃO**

Head Office

Caixa Postal 8
38180 Araxá, MG - Brazil
Phone: (034) 661.1544 Telex: (034) 3355 CBMM BR

Branch Offices

BRASIL

Companhia Brasileira de Metalurgia e Mineração
Rua Padre João Manoel, 923 - 9º andar
01411 - São Paulo, SP - Brazil
Phone: (011) 881.7100 Telex: (011) 25683 CBMM BR

EUROPE

Niobium Products Company GmbH.
Wagnerstrasse 4
D-4000 Düsseldorf - 1 West Germany
Phone: (211) 35.3404 Telex: (41) 8587006 NPC D

NORTH AMERICA

Niobium Products Company Ltd.
440 Park West Bldg. Two
Cliff Mines Road
Pittsburgh, PA 15275 - U.S.A.
Phone: (412) 787.9620 Telex: (230) 90.2936 NPC-PGH

ORIENT

CBMM Internacional
Akasaka Brighton Bldg., 5th Fl.
5-2, Akasaka 1 - Chome. Minato-Ku
Tokyo 107 - Japan
Phone: (3) 586.3921 Telex: (72) 26616 CBMM J

DEVELOPMENTS IN NIOBIUM STEELS FOR LINEPIPE APPLICATIONS^(*)

F. Heisterkamp, Niobium Products Company GmbH
B. Bergmann, Dillinger Hütte
H. Stuart, Niobium Products Company Ltd
L. Chaussy, Dillinger Hütte

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Contents

1 — Introduction	1
2 — Results	2
2.1 — <i>Mechanical properties</i>	2
2.2 — <i>Weldability</i>	7
3 — Summary and Conclusions	9
4 — References	10

TABLES AND FIGURES

Table I — Comparison between Dillinger X65 — X70 line pipe composition and other compositions currently produced by other manufacturers	3
Table II — Composition and processing schedule for present rolling trials	3
Table III — Properties of 17.5 mm X70 plate made from niobium-titanium steel (mean values of 2000 t plate production	6
Table IV — Implant test results of low carbon-niobium-titanium steels	6
Fig. 1 — Effect of finish rolling temperature on the mechanical properties of a 0.08% carbon steel having various niobium contents	11
Fig. 2 — Mechanical properties of steels with varying niobium content	12
Fig. 3 — Properties of 17 mm plate standard grade X70 Dillinger Hüttenwerke	13
Fig. 4 — Hydrogen entry rate for various steels	14
Fig. 5 — Mechanical properties of steels from present trials	15
Fig. 6 — Isotoughness curves (50% FATT) for different rolling temperatures and niobium contents	16
Fig. 7 — Correlation between niobium-titanium content and finish rolling temperature for 20 mm pipe plate	17
Fig. 8 — Vector diagram illustrating general effects of niobium on as-welded toughness	18
Fig. 9 — Effect of weld cooling rate on HAZ toughness in niobium steels	19
Fig. 10 — Weldment properties of uo-pipe (th. 15 mm)	20
Fig. 11 — Weldment properties of X70 pipe made from: a) 0.5% Ni — 0.13% Nb — Steel b) 0.035% Nb — 0.05 to 0.09% Ti — Steel	21
Fig. 12 — Simulated girth welding of 25 mm X70 niobium-titanium plate	22

ABSTRACT — *Current niobium containing steels being produced for line pipe applications, developed over the last ten years, demonstrate an excellent response to heavy deformation at low temperatures, which results in an optimum balance of yield strength and toughness. However, it has long been recognized that excessive use of controlled rolling involves production penalties and contributes to the characteristic anisotropy present in rolled products. Thus, changes in rolling procedures would be desirable if they minimized delays or reduced directionality thereby resulting in further improvement of secondary properties, such as through thickness ductility and hydrogen induced cracking (HIC) tendency in sour gas environments.*

Present steel development is focused on transformation strengthening or increased precipitation hardening. Coupled with the trend to even lower carbon (<0.025%) and nitrogen level: (20 ppm), new alloy designs including titanium have become feasible.

This paper describes new steel types, which have been developed to embody some of the above variables. A significant feature of the steels is their chemistry-processing balance, comprising a critical niobium/titanium content in conjunction with possibilities of processing at relatively high finish rolling temperatures to produce a homogeneous equiaxed ferrite microstructure.

1 - Introduction

During the last two decades the technology of controlled rolled microalloyed steels has been refined to a high degree by the world's major steel producing companies. More recently, this technology has been adopted by the developing nations as they seek to optimize their steel needs.

However, it has long been recognized that the use of controlled rolling involves mill production penalties and development of banded and textured ferrite which contributes to the characteristic anisotropy present in rolled products. The intensity of such anisotropy is accentuated by deformation in the ferrite-austenite two-phase region. Thus, avoiding this procedure might result in further improvement of secondary properties and contributes to the overall attractiveness of these tough, high-strength steels.

Moreover, new steelmaking technologies provide excellent means for mass production of low-carbon, low-nitrogen steels that offer new possibilities for steel development and make new alloy designs both feasible and desirable: e.g. niobium-titanium, niobium-titanium-vanadium and niobium-boron types having optimized ratios of

precipitation and grain refinement even when higher temperature rolling regimes are used.

In recent years some attempts have been made to produce microalloyed steels having optimized microstructures by finishing the rolling reduction at temperatures well above the austenite decomposition temperature. These studies have largely investigated the effect of increasing the volume fraction of precipitate particles at high temperatures in austenite, as proposed by Gray ⁽¹⁾, and the composition optimization with regard to chromium, boron and molybdenum content. Kozasu reported in 1975 ⁽²⁾ that in a 0.08 percent carbon steel a niobium content of about 0.08 percent was sufficient to allow excellent toughness and yield strength to be developed when a finish rolling temperature of 1000°C was used in the production of 20 mm thick plate (Fig. 1).

In 1975 and 1977 patents were issued in Canada ⁽³⁾ and USA ⁽⁴⁾ respectively, which reported that in the presence of sufficient concentrations of niobium (0.08%), a fine-grained equiaxed ferrite microstructure could be produced even at finish rolling temperatures well above those thought necessary for effective controlled rolling.

More recently, Chilton and Roberts ⁽⁵⁾ showed similar results from their laboratory investigation of the effects of niobium on the microstructure of low carbon steels deformed with a range of finishing temperatures. Their results are in Fig. 2 combined with other available data from our own and various literature sources. With a finishing temperature of 850 to 950°C, low temperature toughness is combined with a strength of 420 N/mm² (X60). If higher strength or even lower transition temperature is required, lower rolling temperatures or additional alloying must be used.

Further investigation ^(6,7) of this alloy concept including addition of 0.5% nickel led to evaluation of a full scale trial heat ⁽⁸⁾. Longitudinally welded pipe (42 inch diameter x 15-20 mm) and 16 mm thick spiral pipe, possessed X70 strength levels with excellent low temperature toughness (85% DWTT FATT < -50°C) despite rolling in the 800°C temperature range.

The purpose of this paper is to report on additional rolling studies used to develop correlations among composition, finishing temperature, strength and toughness.

2 - Results

2.1. Mechanical properties

Line pipe grades currently produced by Dillinger Hütte are representative of current world technology. These steels contain niobium and vanadium. The compositions are shown in Table I, where

TABLE I - Comparison between Dillinger X 65 - X 70 linepipe composition and other compositions currently produced by other manufacturers. Compositions refer to 15 to 20 mm plate.

PRODUCER	C	Mn	Si	S	Nb	V	OTHERS
<i>Dillinger Hütte</i>	0.09	1.55	0.30	0.003	0.03	0.07	—
<i>Kaiser Steel</i>	0.07	1.55	0.25	0.010	0.04	0.05	—
<i>USS</i>	0.10	1.40	0.23	0.010	0.025	0.08	Cu: 0.22, Ni: 0.21
<i>Bethlehem Steel</i>	0.08	1.68	0.26	0.005	0.04	0.08	—
<i>Italsider</i> a)	0.08	1.55	0.25	0.007	0.04	0.08	—
b)	0.08	1.55	0.25	0.007	0.04	—	Cr: 0.20
<i>NKK</i>	0.08	1.57	0.26	0.003	0.03	0.08	—
<i>Kawasaki Steel</i>	0.07	1.59	0.26	0.003	0.035	0.07	—

TABLE II - Composition and processing schedule for present rolling trials

COMPOSITION

Base : 0.08% C, 1.50% Mn, 0.30% Si, 0.0140% N
 Niobium: 0%, 0.03%, 0.058%, 0.115%, 0.146%, 0.178%

PROCESSING

$T_{\text{Reheating}}$ 1240°C
 Thickness 200 mm
 Thickness, intermediate 100 mm
 $T_{\text{F, Start}}$ 985°C, 915°C, 860°C, 810°C
 $T_{\text{F, Last pass}}$ 920°C, 850°C, 800°C, 750°C
 Thickness 20 mm

a comparison is also made with other niobium-vanadium grades produced by other major pipe producers. In all cases conventional low temperature rolling practices are applied. Strength and toughness properties of Dillinger Hütte's standard grade X70 production are shown in Fig. 3. Application of controlled rolling and calcium treatment result in excellent toughness properties. The reported high plate strength is necessary to achieve X70 pipe properties after pipemaking Bauschinger losses.

The above steel is modified when intended for use in sour gas environment. The modifications follow the lines of modern thinking by basically seeking means for preventing both the entry of hydrogen into the steel and the initiation of cracks. The steel has a reduced manganese content and increased nickel and copper levels. The low sulfur content combined with sulfide shape control prevents crack initiation, whereas the reduction of manganese decreases segregation and the occurrence of banded structures.

The effects of these compositional changes on British Petroleum diffusible hydrogen measurements are illustrated in Fig. 4. Clearly, the "low manganese, plus copper and nickel" philosophy in combination with sulfide shape control allows a much lower hydrogen entry rate compared to conventional alloy levels.

As outlined earlier, from various points of view it is desirable to investigate alternatives to current production methodology in order to gain a higher degree of plate mill flexibility. Therefore, a series of trials was carried-out on Dillinger Hütte's heavy plate mill, in which we investigated the utility of higher temperature processing. The investigation was carried-out using steels having the compositions shown in Table II. Plates 20 mm thick were rolled with finishing temperatures of 750 - 920°C from reheating temperatures of 1240°C. Due to a special "embedded ingot" technique all the steel compositions were rolled with exactly the same rolling schedule.

The mechanical properties of these steels are summarized in Fig. 5. Several features of significance are apparent: firstly, by raising the niobium content to the region of 0.07 - 0.08% the strength of the material finished at greater than 850°C reaches 430 MPa. Finishing at 720°C produces 470 MPa. For all schedules, as the niobium content increases the toughness progressively improves, to yield a 50% FATT of about -80°C with 0.1% niobium and finish rolling at 850°C.

An alternative perspective of the effect of finishing temperature on transition temperature as a function of niobium content is shown in the isotoughness graph in Fig. 6. It is evident that, for the achievement of a given toughness level, increasing niobium contents allow relaxation of rolling schedules (higher finishing temperatures). Under these circumstances niobium is acting mainly as a toughening element. To reach higher strength levels, additional measures have to be adopted. Since the trends in steelmaking are favouring low carbon

and low nitrogen one must select precipitation strengthening elements. In this context additional trials have been carried out with titanium in the system low carbon-manganese-niobium having compositions close to stoichiometry to maximize the precipitate volume fractions.

The additional trials in the system niobium-titanium had various objectives:

- I. To evaluate the strengthening effect of titanium at low finishing temperatures and conventional niobium contents.
- II. To carry out controlled rolling with a higher finishing temperature, using slightly increased niobium content and compensating for the yield strength loss by titanium additions.
- III. To extend the above case by evaluating the effect of further increased niobium contents on toughening of niobium-titanium steels.
- IV. To develop a steel for finish rolling $\geq 800^{\circ}\text{C}$ thereby simultaneously reducing rolling mill load, increasing rolling capacity, minimizing texture, reducing the degree of "separations" and increasing shelf energy.

In agreement with previous investigations^(9, 10, 11) it was found that only titanium as titanium carbide could be considered as a strengthening agent. However, titanium may strengthen indirectly by tying up the nitrogen at high temperatures ($> 1000^{\circ}\text{C}$), thus preventing the premature precipitation of niobium nitrides in austenite. When not prevented, this process will normally limit subsequent precipitation hardening potential after transformation to ferrite. The effect of TiN formation in increasing yield strength (through increased NbC precipitation) was found to be about 20 N/mm^2 . Additional toughness benefits result from titanium nitride formation by virtue of minimizing mobile nitrogen in the as-rolled steel and controlling austenite grain coarsening at high temperatures.

In all trials the strengthening effect of titanium at an average nitrogen content of 80 ppm was found to be 20 N/mm^2 per 0.01% Ti eff^(*). This is in good agreement with Thyssen data^(9, 10) of 22 N/mm^2 and within the range of Japanese investigations⁽¹¹⁾ of 16 to 35 N/mm^2 for various rolling schedules.

The results of all aforementioned mill trials are summarized in Fig. 7. The starting point A represents a heavy wall (20 mm) low carbon steel containing a conventional niobium addition (0.03% Nb) reheated to 1240°C and rolled with a finishing temperature of 720°C . Yield strength is reduced and transition temperature is increased, when the finish rolling temperature is raised to 800°C (point B). However, as shown in the present investigation, yield strength increases

(*) Ti available to combine with carbon.

TABLE III - Properties of 17.5 mm X 70 plate made from niobium-titanium steel (mean values of 2000t plate production)

COMPOSITION IN %					
C	Si	Mn	S*	Nb	Ti _{tot.}
0.065	0.45	1.48	0.003	0.03	0.048
R _{p0.5}				540 N/mm ²	
R _m				605 N/mm ²	
Yield ratio (plate)				0.89	
A ₂ ^r				38%	
CVN, 0°C				190 Joule	
CVN 50% FATT				— 100°C	
85% BDWTT				— 55°C	

* Sulfide shape control

TABLE IV - Implant test results of low carbon-niobium-titanium steels

TEST CONDITIONS

Welding material: a) Cellulosic electrode Cel 90
 b) MAG with Fluxofil / CO₂
 Heat input : 7-8 kJ/cm (without hot pass)
 Applied stress : 490 N/mm²; 6mm dia specimens

TEST RESULTS

a) Cel 90

Steel-No.	%C	%Mn	%Nb	%Ti	C _{eq}	T(min. preheat.)
1	0.09	1.53	0.034	—	0.36	70°C
2	0.06	1.46	0.031	0.05	0.31	60 - 70°C
3	0.06	1.50	0.030	0.09	0.32	60 - 70°C
4	0.06	1.45	0.032	0.12	0.31	60 - 70°C

b) MAG

3 } Crack free specimens without any
 and } preheating even at an applied
 4 } stress of 580 N/mm²

to 430 N/mm² with a remarkably improved toughness when niobium content is raised to 0.08% (point C).

If titanium is added at this point, yield strength can be raised further and almost independent of finish rolling temperature. Since in this context titanium is also adding to grain refinement, a yield strength of 490 N/mm² can be achieved without impairing the toughness at effective titanium additions of 0.03 percent (point D). Slight changes in rolling practice help to maintain the toughness level.

A further increase of effective titanium to 0.06 percent adds 54 N/mm² to yield strength with a 10°C higher transition temperature (point E). Alternatively, similar strength levels can be achieved using leaner compositions, if traditional low temperature rolling is applied (point F). Additional improvement of low temperature toughness can be obtained in this alloy system by increasing niobium level even when finish rolling at low temperature (points G and H).

Vanadium precipitation has also been investigated and even though it plays a similar strengthening role, the strength increase per unit addition is rather small. Furthermore, it should be noted that effectiveness of vanadium strengthening diminishes as carbon and nitrogen contents are reduced as outlined in a recent Japanese overview⁽¹²⁾.

Based on these investigations more than 2000t of low carbon-manganese-niobium-titanium steel have been produced at Dillinger Hütte and rolled to 17.5 mm thick X70 pipe plate. In this case low temperature rolling practice was applied and the properties (mean values) are presented in Table III. The results demonstrate that the toughness of the product is suitable for the most severe arctic conditions.

2.2 Weldability

The steels described in the present paper in some cases contain two or three times the niobium concentration customarily found in pipeline steels. Moreover, some of the steels have a content of up to 0.09% Ti total. Consequently, the weldability of these steels needs to be considered. Niobium and titanium have no significant effects on general aspects of weldability such as heat affected zone hydrogen cracking, liquation cracking or weld metal solidification cracking and thus, our main considerations of weldability refer to the effect of these elements on weld heat affected zone (HAZ) toughness and weld metal toughness.

There has been considerable confusion concerning the role of niobium in weldments at the levels of present interest, mainly because of the complexities of the different welding processes and microstructures in the absence of niobium. Fortunately, the field has

been comprehensively reviewed by Kirkwood in a recent paper⁽¹³⁾. The major factor of importance is the ability of niobium in solution in austenite to depress the austenite to ferrite transformation temperature. Thus, as with other alloying elements, the effect of niobium depends on what the resultant microstructure would have been in the absence of niobium. Clearly, therefore, niobium could be beneficial or harmful. To illustrate this for weld metal toughness Dolby⁽¹⁴⁾ constructed a vector diagram based on earlier considerations by Garland and Kirkwood⁽¹⁵⁾ and this diagram is reproduced in Fig. 8. Similarly, HAZ toughness can be improved or impaired by niobium additions. Niobium can refine the austenite grain size (which is beneficial) by preventing grain coarsening especially with low heat inputs and fast cooling rates. Alternatively, with high heat inputs and slow cooling rates, the harmful effects of strengthening through precipitation may dominate. These effects are illustrated in Fig. 9 taken from work conducted by Rothwell⁽¹⁶⁾ using instrumented Charpy-V-Notch, CVN, tests. French investigations⁽¹⁷⁾ show a similar influence of cooling rate and documented the transition from beneficial to harmful effects at a ΔT 800/500°C of about 20 seconds.

Although harmful effects can be projected from increased levels of niobium, satisfactory means of overcoming this have been developed. In particular, for weld metal toughness, the application of appropriate wire/flux combinations, that produce a predominantly acicular ferrite microstructure, has been successful. Two new state of the art reviews^(18, 19) on the subject have been published recently.

Similar considerations are relevant for titanium additions especially during weld reheat-treatments (stress relieving) except that titanium nitride formation may improve weldment properties through reducing dissolved nitrogen content and refining HAZ microstructure.

With the above considerations in mind, plates with various niobium and titanium contents, as discussed before, have been welded into pipe. Composition and weldment properties of 15 mm X70 pipes with 0.13% Nb are shown in Fig. 10. Using consumables that provide for an acicular ferrite weld metal microstructure and applying conventional three-wire submerged arc pipe welding conditions, good weld metal toughness down to -60°C was achieved in conjunction with good heat affected zone toughness down to -40°C .

Weldment properties of niobium-titanium steels, obtained in the current study, are shown in Fig. 11 together with data from Japanese pipe⁽¹¹⁾, produced from low carbon 0.07 – 0.09% titanium steels. The niobium-titanium pipe was welded using a three-wire method and L70/L761 wire/flux combination at 25/30 kJ/cm heat input. The toughness of the weld metal and heat affected zone is similar to conventional X70 pipe weldments. Small differences in toughness in the reference steel may be related to the specimen position, which in our case is in the first, less tough bead.

In addition to these results the performance of new pipe steels during field welding is important, in particular for submarine pipelines. Extensive implant tests on steels with various titanium contents have been carried out. The test conditions and results are shown in Table IV. The susceptibility to cold cracking of this steel type is low even at high (0.12%) titanium contents.

The preheating temperature was raised until 2 crack-free results were achieved. The minimum preheating temperature of 60 to 70°C is in line with existing X70 steels. No preheating is necessary in the absence of hydrogen (automatic welding). Nevertheless, simulated girth welding was carried out on 25 mm plate (0.03% Nb, 0.09% Ti) without preheating (Fig. 12). At the lowest applied heat input of 5 kJ/cm maximum hardness in the area of the last bead was 290 to 310 HV5. The weldment properties are good and in line with obtainable toughness levels for the applied technology.

The present data have shown, with respect to welding, that steels containing both niobium and titanium can be used in most normal applications as a replacement for conventionally alloyed steel, provided that welding procedures and consumables are properly specified.

3. Summary and Conclusions

The production of high strength low alloyed line pipe steels (grade X70) and steels for sour gas application at Dillinger Hütte are representative of current world technology.

However, from various points of view, the development of new alloy designs and rolling concepts was desirable. Investigations in this context produced the following results:

- At low carbon contents, titanium proved to be a strong precipitation hardening element. With optimized rolling schedules, good strength to toughness ratios were obtainable in niobium/titanium steels.
- Excellent toughness levels were achieved, when the niobium content in such steels was raised to somewhat higher levels (0.08%).
- With a balanced niobium and titanium content, good strength to toughness ratios could be secured even with a relaxed rolling schedule (F. R. T. ~ 800°C).

Fabrication and welding of pipes from these steels demonstrated that even in the presence of high concentrations of alloying elements niobium and/or titanium, the application of appropriate submerged arc welding procedures and consumables produced good pipe weldment properties. Field weldability was tested and results are in line with those obtainable on standard pipe grades.

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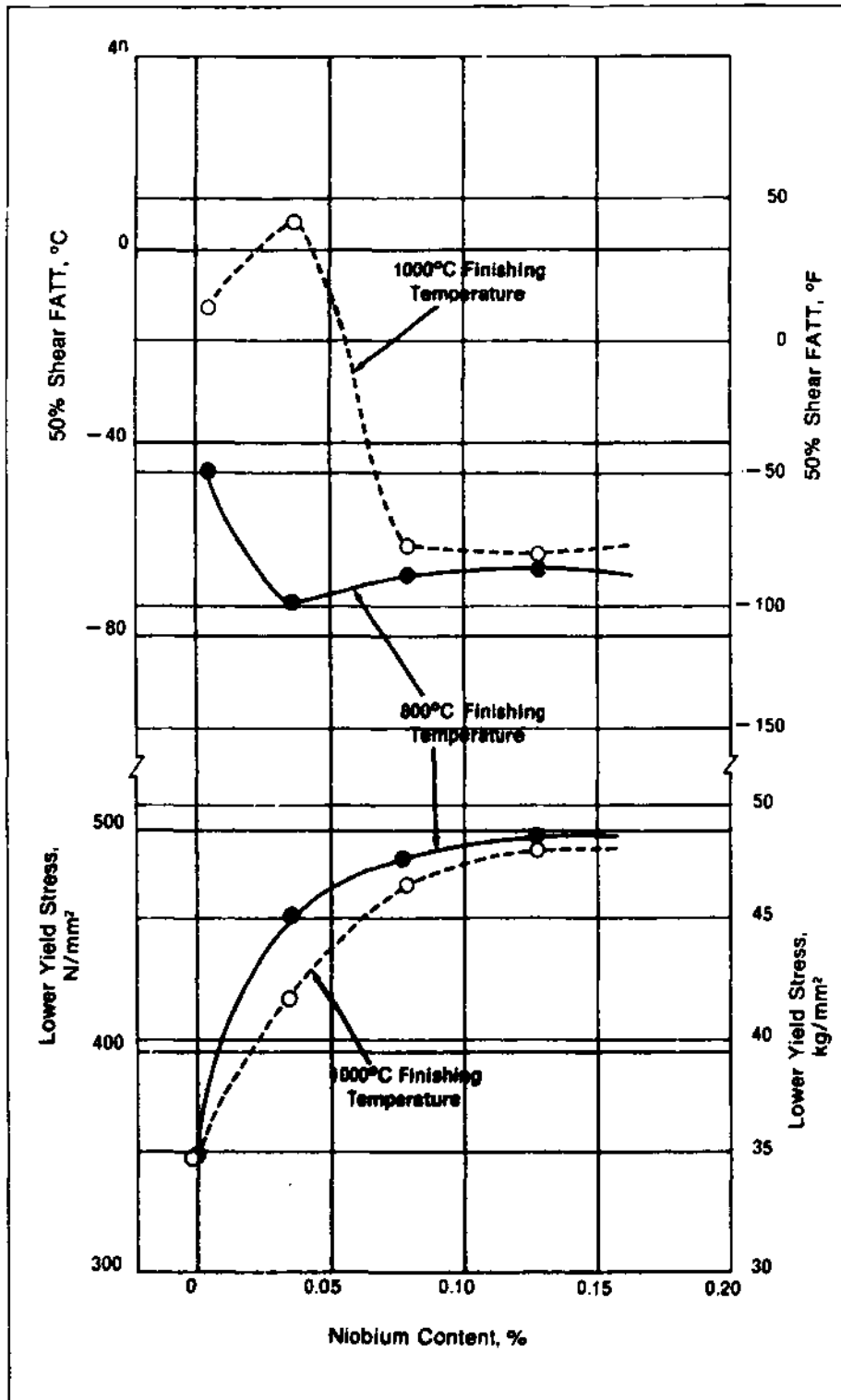


Fig. 1 — Effect of finish rolling temperature on the mechanical properties of a 0.08% carbon steel having various niobium contents (after ref. 2).

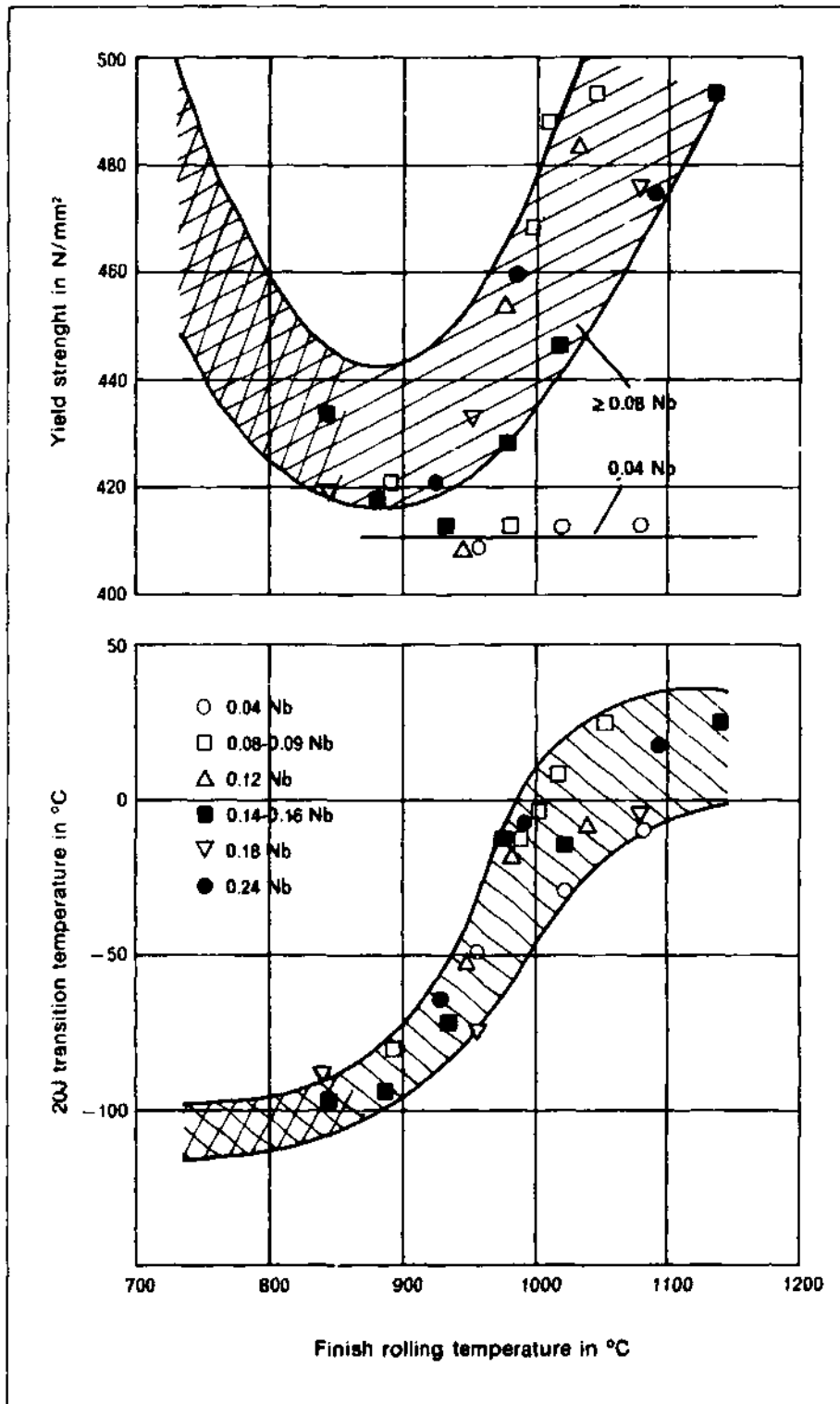


Fig. 2 — Mechanical properties of steels with varying niobium contents (after ref. 5 and various other and own results).

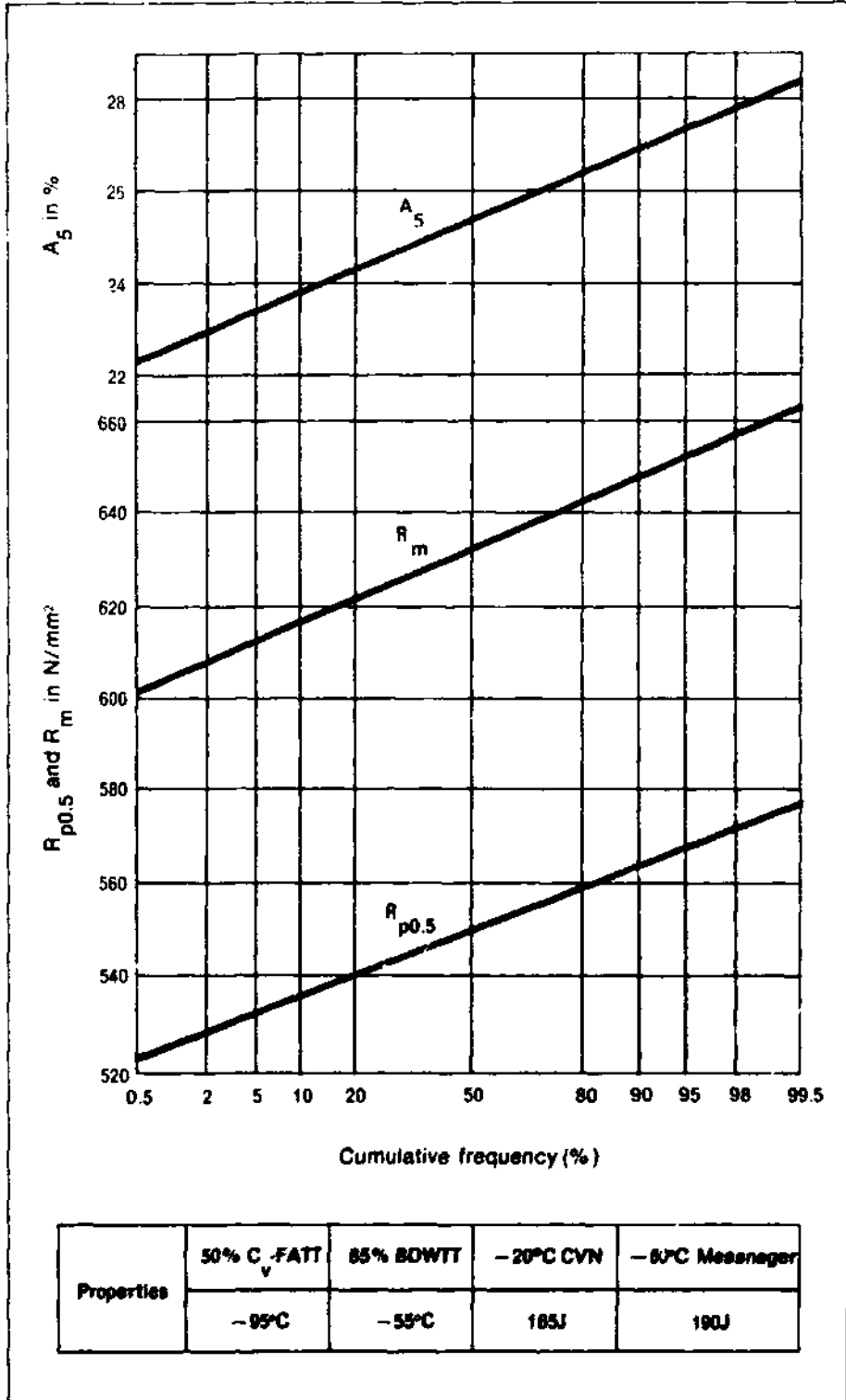


Fig. 3 — Properties of 17 mm plate Standard grade X 70 Dillinger Hüttenwerke (transverse).

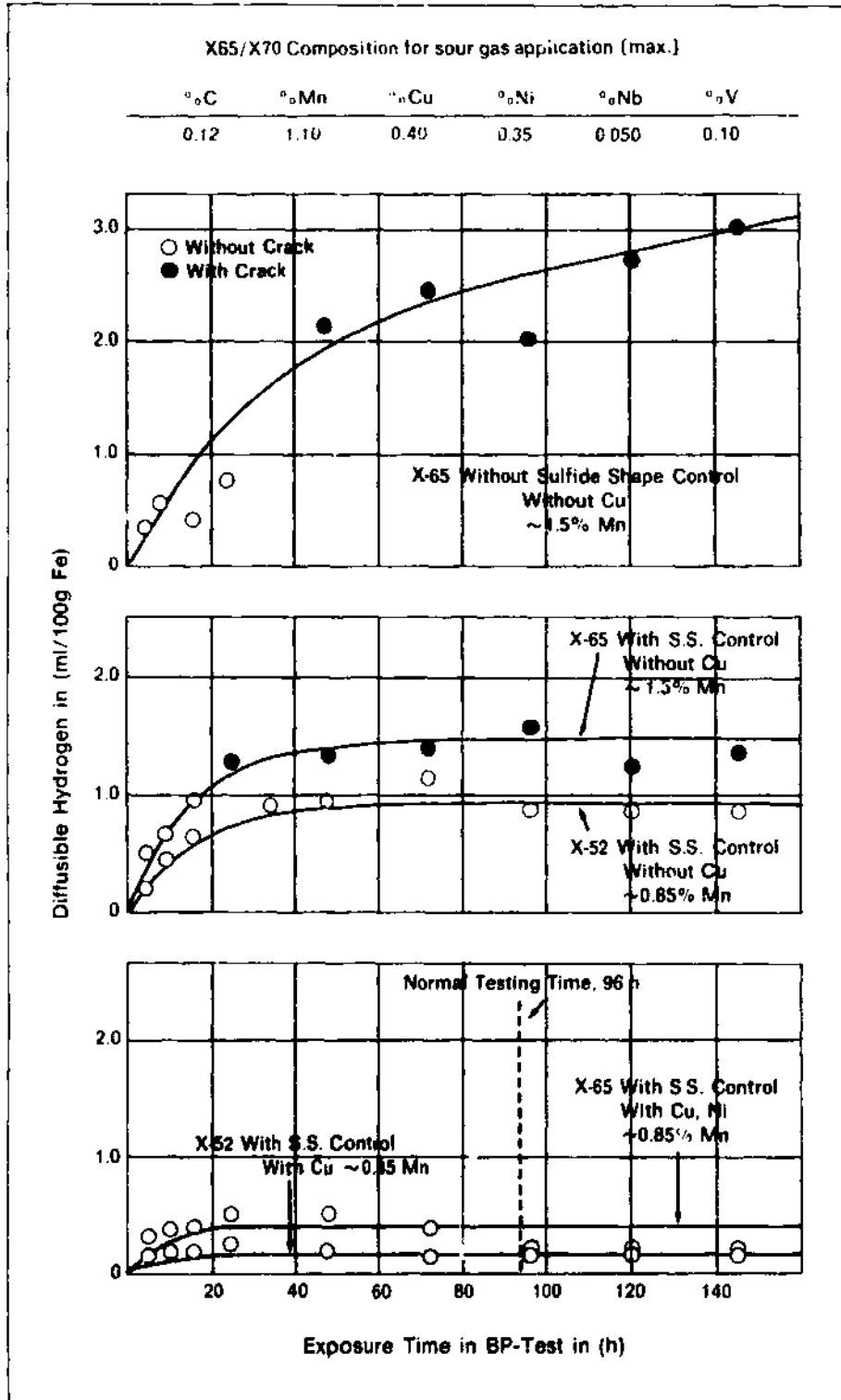


Fig. 4 — Hydrogen entry rate for various steels.

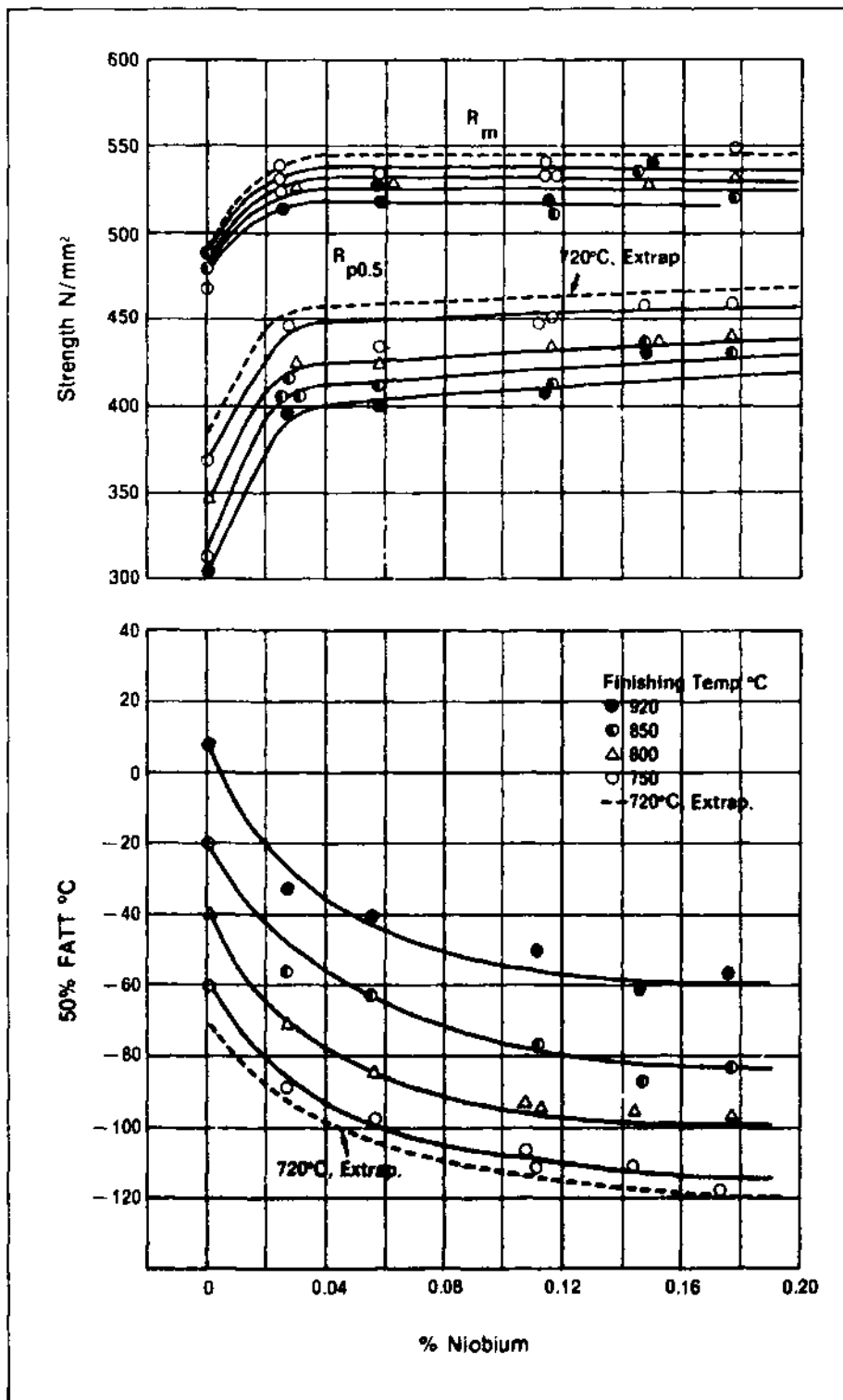


Fig. 5 — Mechanical properties of steels from present trials.

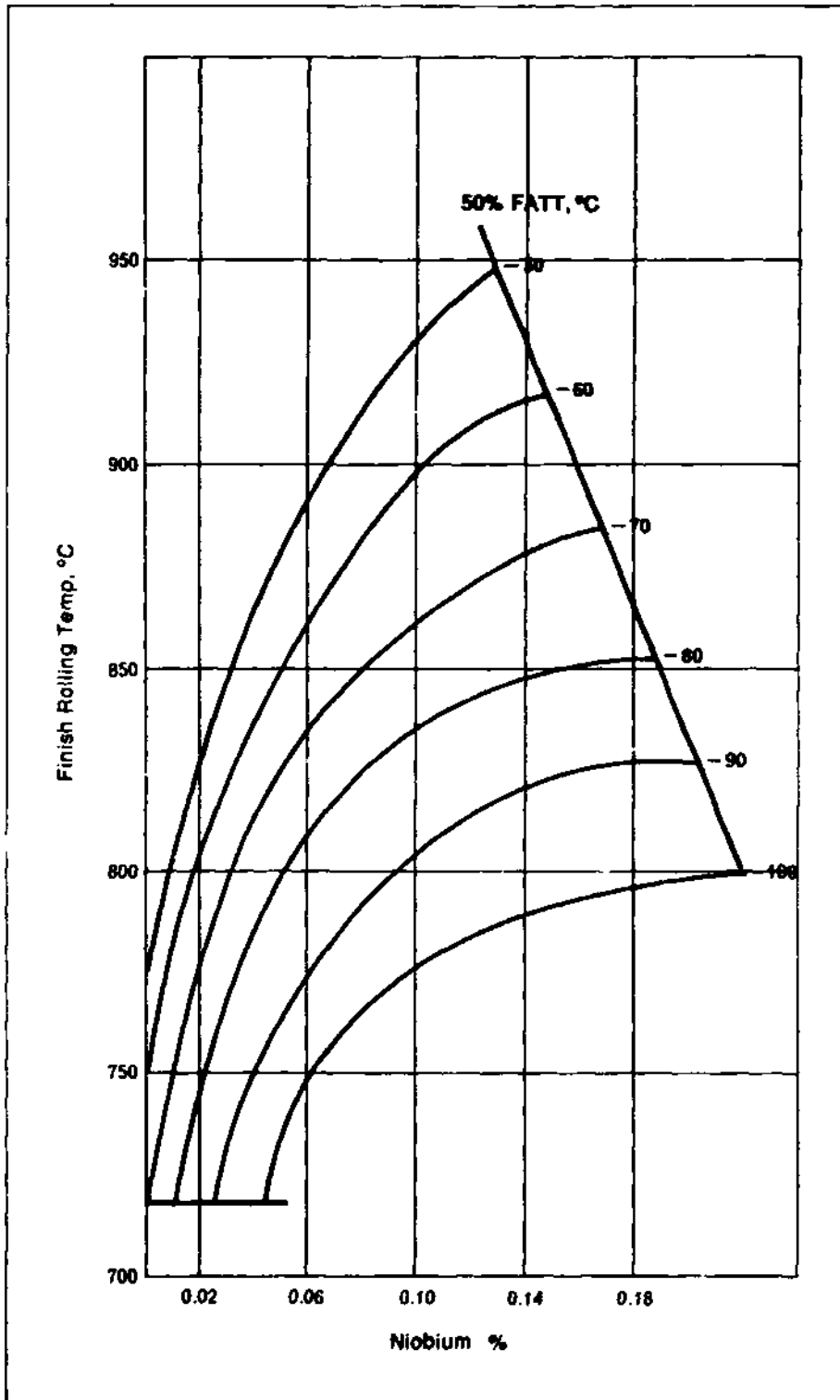


Fig. 6 — Isotoughness curves (50% FATT) for different rolling temperatures and niobium contents.

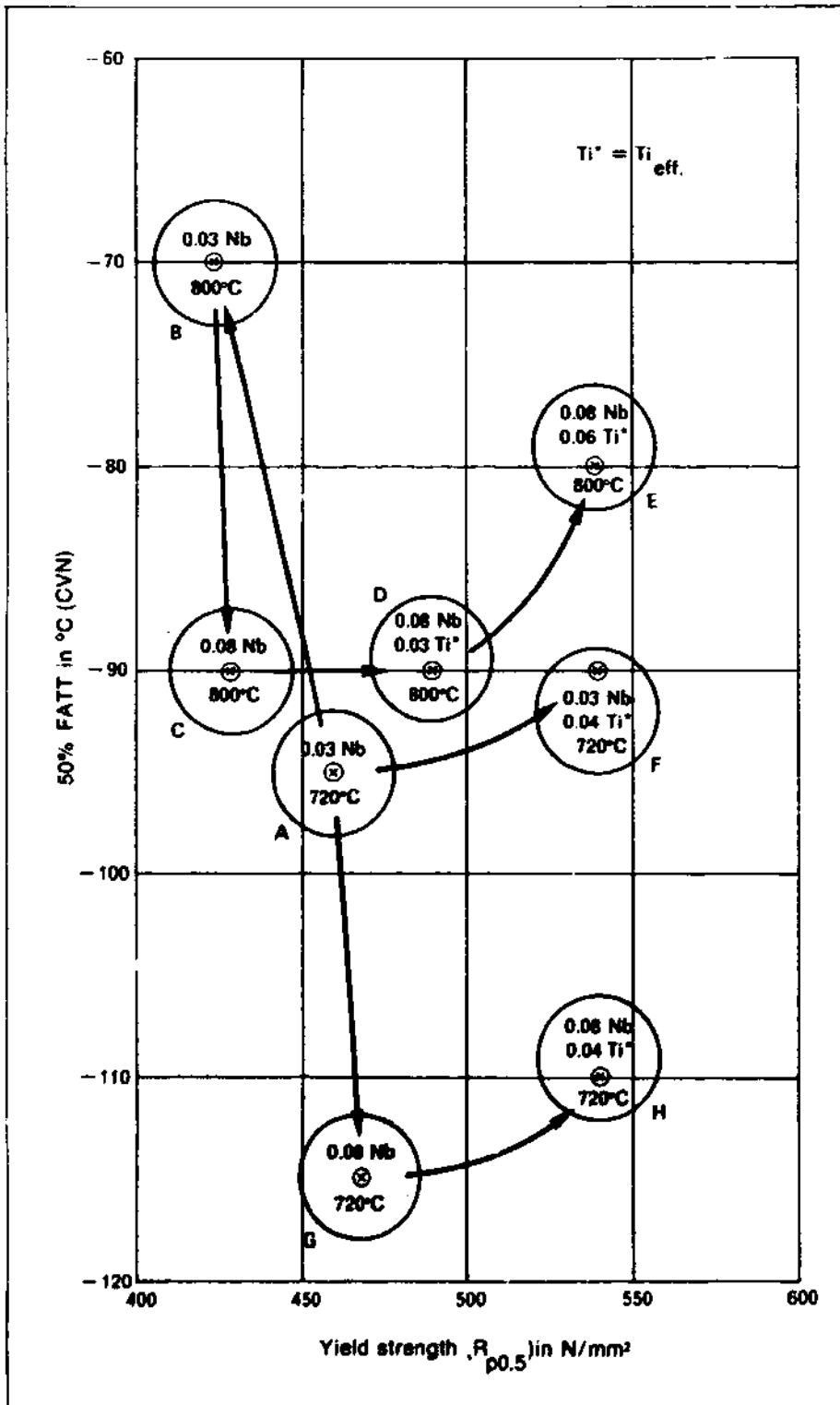


Fig. 7 — Correlation between niobium-titanium content and finish rolling temperature for 20 mm pipe plate (base composition: 0.08% C; 1.5% Mn; 0.30% Si).

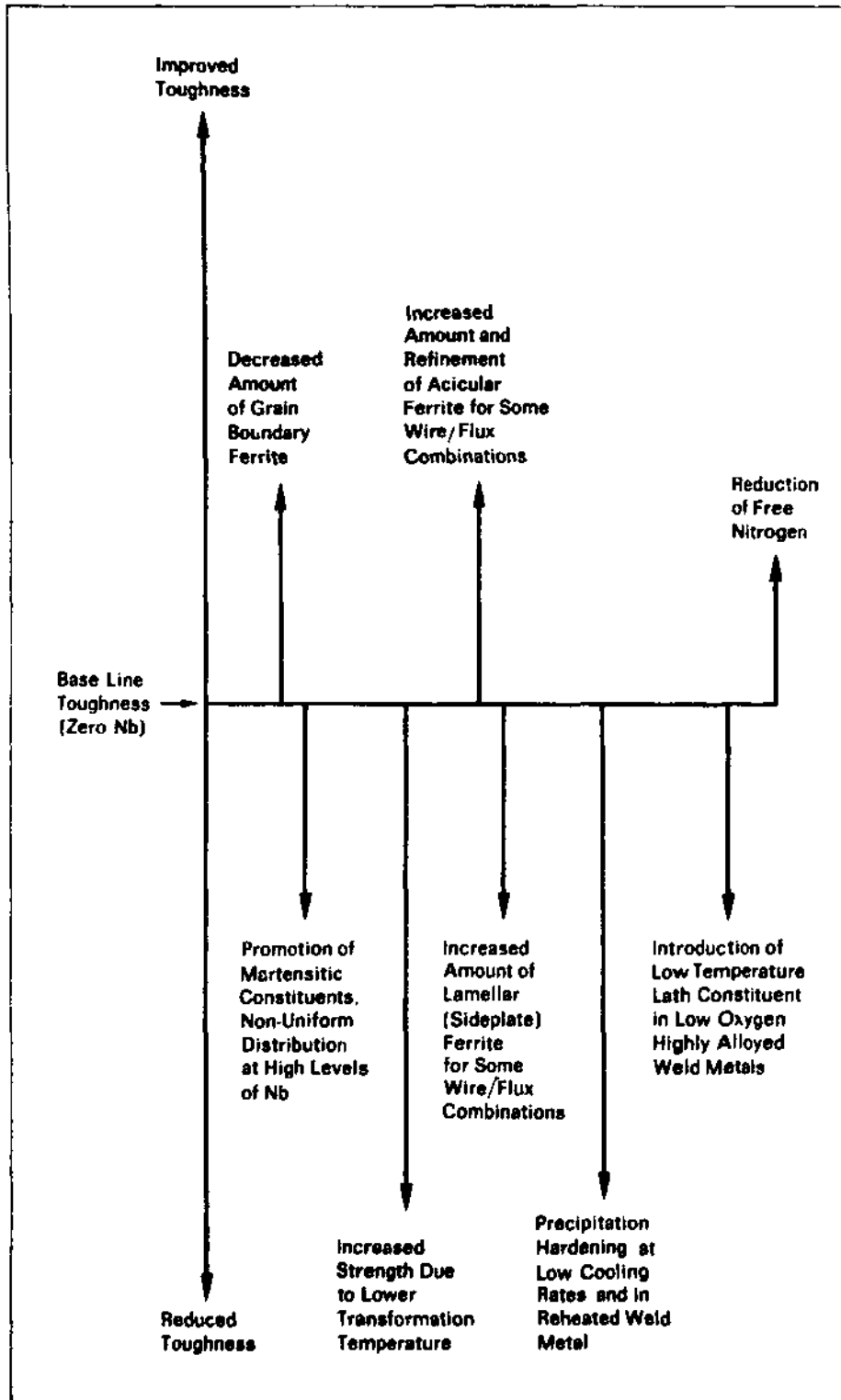


Fig. 8 — Vector diagram illustrating general effects of niobium on as-welded toughness (after ref. 14).

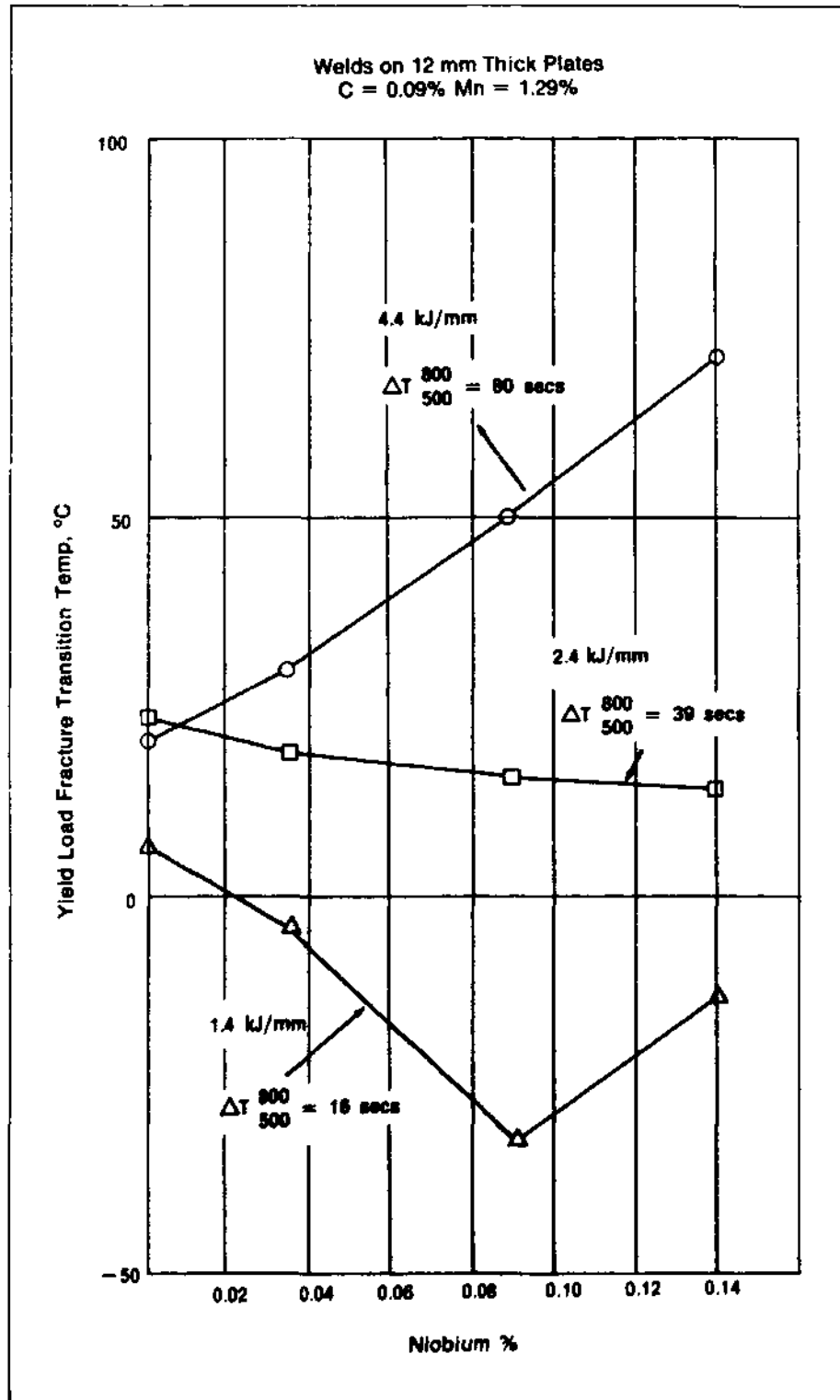
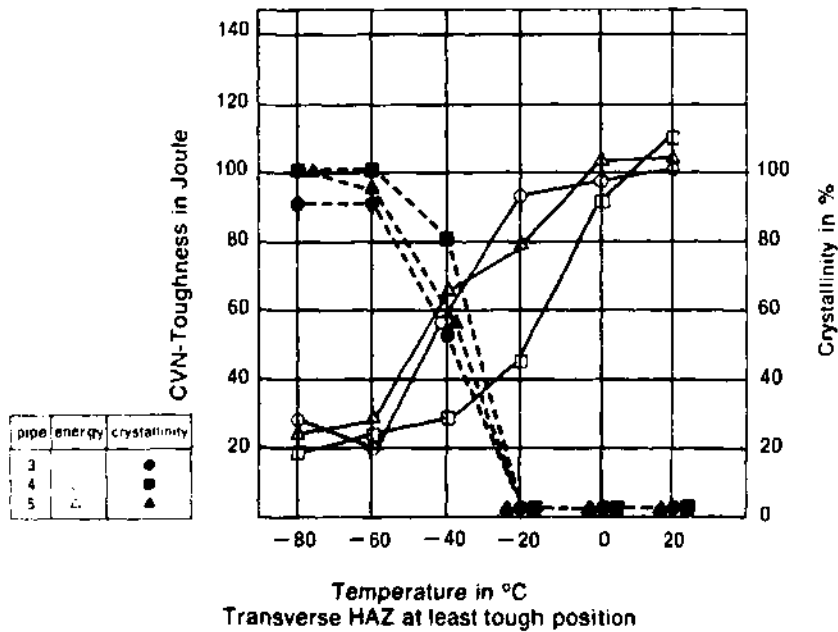
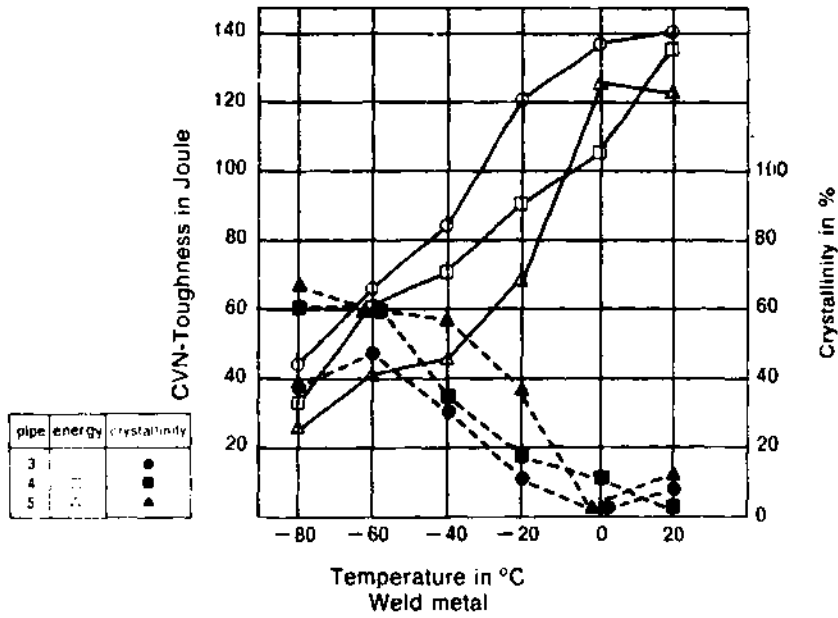


Fig. 9 — Effect of weld cooling rate on HAZ toughness in niobium steels (after ref. 18).

Consumables: S2MoTi and a fused, Boron containing, amphoteric flux; H.I.: 29 kJ/cm



STEEL COMPOSITION: 0.08% C, 0.32% Si, 1.51% Mn, 0.54% Ni, 0.13% Nb

Fig. 10 — Weldment properties of uo-pipe (th. 15 mm).

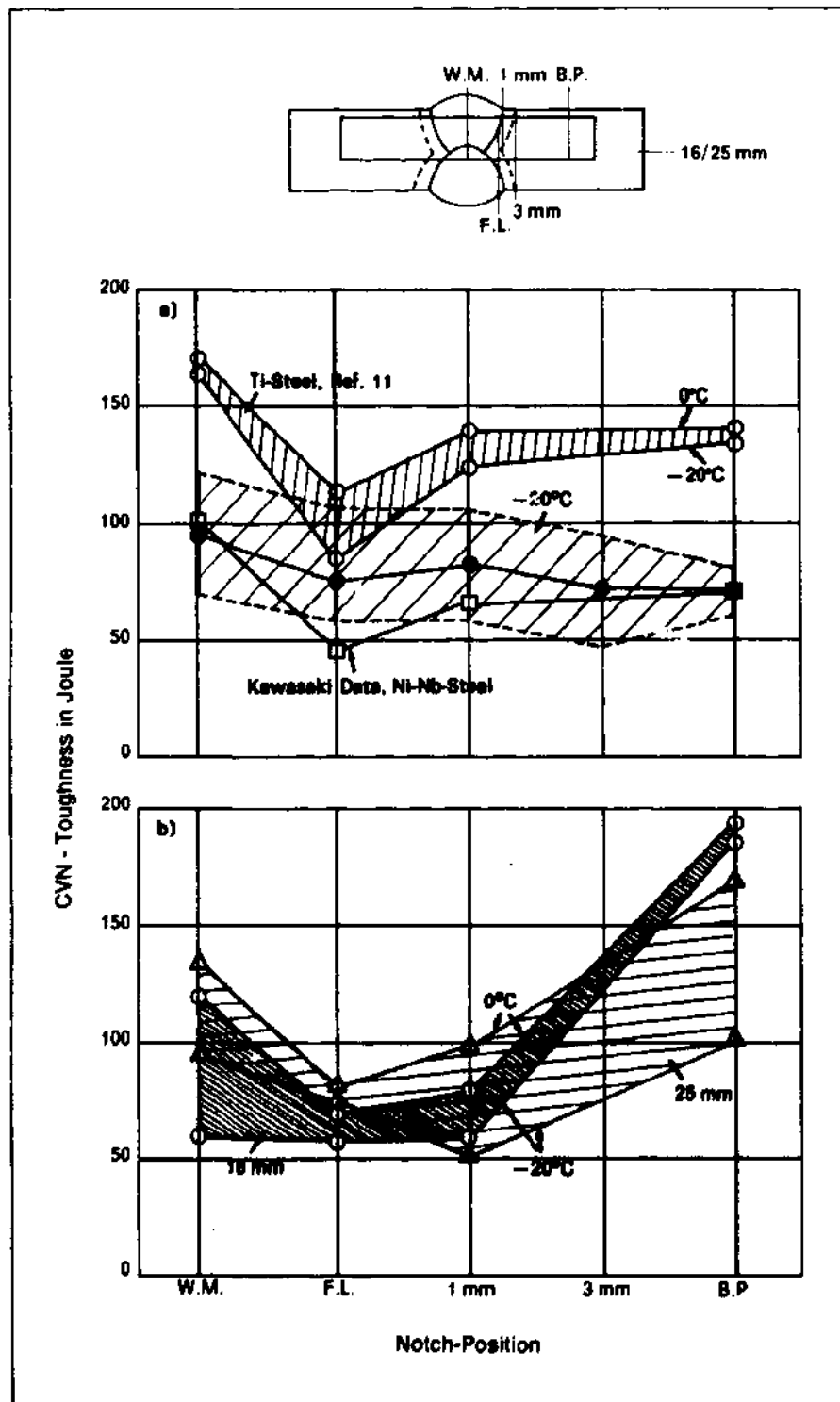


Fig. 11 — Weldment properties of X 70 pipe made from:
 a) 0.5 Ni — 0.13% Nb — Steel
 b) 0.035% Nb — 0.05 to 0.09 Ti-Steel.

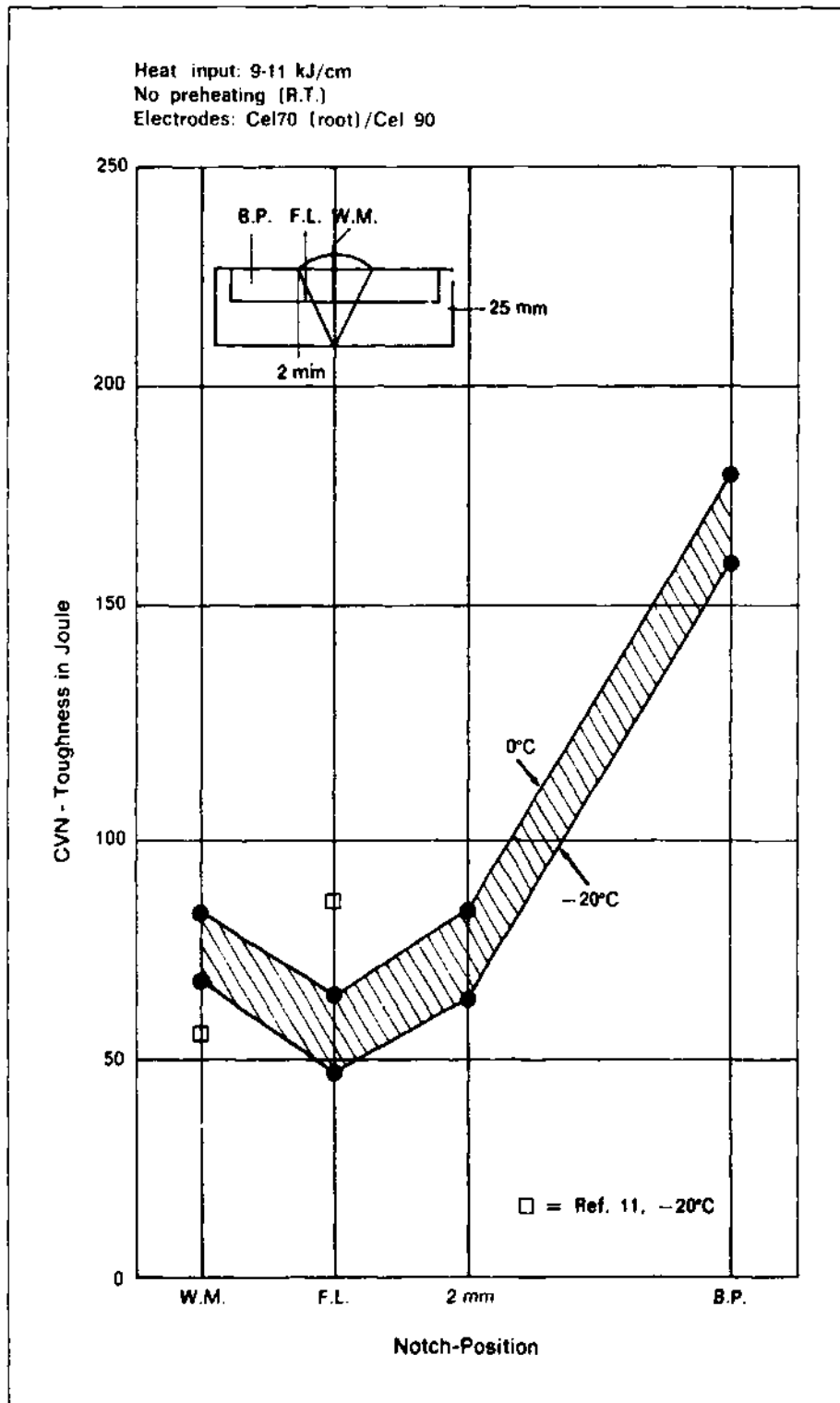


Fig. 12 — Simulated girth welding of 25 mm X 70 niobium-titanium plate.



**COMPANHIA BRASILEIRA DE
METALURGIA E MINERAÇÃO**

CBMM is a Brazilian Corporation, engaged in mining, beneficiation and industrialization of the pyrochlore reserves located in Araxó, Minas Gerais. These reserves amount to 460 million tons of ore with a Nb_2O_5 concentration of 2.5%. With a fully integrated operation, CBMM has amongst its main products standard and vacuum grade FeNb, NiNb and Nb_2O_5 . Aiming at the development of new applications for niobium and at improving available technologies, CBMM maintains a very active R & D program which is being carried out in universities and research centres both in Brazil and abroad. The Niobium Information Centre (CITEN) located in São Paulo, is a cornerstone of that system - its mission is the dissemination of technical information about niobium.