

# Determination of the ductile-to-brittle transition temperature of NIOMOL 490 K steel welded joints

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## ABSTRACT

This paper is dedicated to the ductile–brittle transition behaviour of the microalloyed structural steel NIOMOL 490 K. This steel grade is used for welded pressure vessels subjected to dynamic loads and operating at sub-zero temperatures. Therefore, it must have an acceptable toughness. Due to its importance for the safety assessment of pressure vessels, a characterization of this steel was carried out using the Charpy V-notch impact test in the temperature range between  $-60\text{ }^{\circ}\text{C}$  and  $+60\text{ }^{\circ}\text{C}$ . The notches were located in parent material, heat affected zone and weld metal. In this paper, the tensile strength properties at ambient temperature and the nil ductility temperature in the temperature range from  $-60\text{ }^{\circ}\text{C}$  to  $+60\text{ }^{\circ}\text{C}$  are presented.

## 1. Introduction

In the transition from pure brittle fracture to the quasi-brittle (ductile–brittle) fracture mode, fracture mechanics investigates not only the phenomenon of fracture, but also the deformation that preceded it. In the case of a ductile–brittle fracture and a ductile fracture, a lot of energy is spent on the development of the plastic deformation that precedes the fracture.

In this respect, the beginnings of fracture mechanics can be found in the first described tests of the mechanical properties of materials and fracture, for example in the 15th century in the works of Leonardo da Vinci.

He described devices for testing the resistance of iron wires of different lengths and came to the conclusion that the resistance decreases with increasing wire length, which is in line with the production possibilities of the 15th century [1].

The concept of the brittle-ductile transition temperature (BDTT) was introduced during World War II. In recent history, the period of frequent failures begins with the introduction of welded constructions. Before and during World War II, 4694 cargo ships – the famous Liberty ships were built in the United States. Fractures of welded structures occur even at low stresses, which made the fractures seemingly inexplicable. Liberty Ships were even broken while anchored in port, Fig. 1, [2,3].

Investigations have shown that these structures exhibited crack-like defects and significant stress concentrations and, in some cases, residual stresses caused by welding. Liberty ship fractures were brittle, with very little or no plastic deformation, Fig. 1(b). It was found that the brittle fracture of parent material and welded joints was caused by low temperatures and a triaxial stress state (plane strain state) that can form at the tip of the sharp notch or crack. The thermal process during welding can also be an important influencing factor.

Fracture resistance of components depends on dimensions, defect, type and extent of, absolute cross-sectional dimensions, test temperature and deformation rate. For most structural materials, a reduction in temperature leads to an increase in strength and a reduction in plasticity. Information on the brittle fracture tendency of material is of particular importance. Brittle fracture is a rapid, catastrophic fracture of the material or the entire structure at stresses below the yield strength of the material and without significant residual deformation. The source of brittle fracture is usually the most highly stressed areas of the material (zone of stress concentration) and defects. Defects include microcracks, microheterogeneity, various microstructural defects, accumulations of dislocations and void.

In general, the impact toughness decreases with decreasing test temperature, as the plasticity or formability of the material also decreases.

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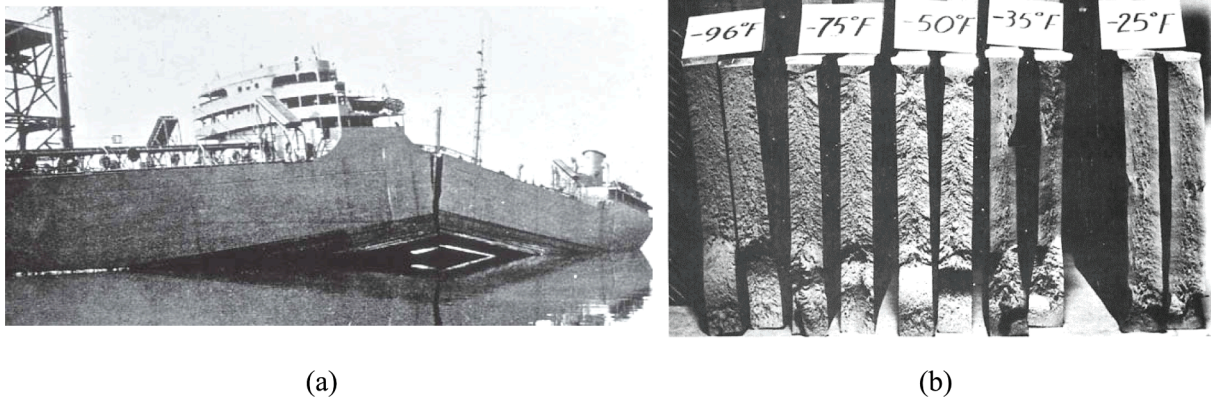


Fig. 1. (a) The Liberty ship S.S. Schenectady. (Reprinted with permission of Earl R. Parker, Brittle Behavior of Engineering Structures, National Academy of Sciences, National Research Council, John Wiley & Sons, New York, 1957.) (b) Ship plates fracture surfaces tested at different temperatures (Reprinted with courtesy of G.F. Vander Voort, Carpenter Technology Corporation) [3].

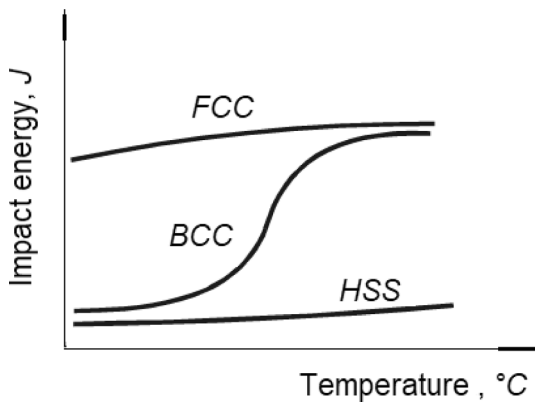


Fig. 2. Impact toughness of structural materials as a function of test temperature [4].

For very ductile metals with face-centered cubic crystal lattice (FCC) such as austenitic steels, silver, copper and nickel alloys, impact toughness decreases gradually and slightly, and these metals are ductile over a wide temperature range. Brittle materials, such as high-strength steels (HSS), glass and ceramics, exhibit low toughness and deformability regardless of the test temperature. Metal alloys with body-

centered cubic lattice (BCC), such as most structural steels, exhibit a characteristic S-shaped curve with a clearly defined transition test temperature, [4], Fig. 2.

The results of numerous experiments on the influence of sub-zero temperatures on welded steel fracture made it possible to draw up diagrams on the dependence of mechanical properties on temperature, as shown for structural steel used in shipbuilding, Fig. 3. The point labeled NDT (Nil Ductility Transition – NDT temperature) determines the temperature below which only brittle fracture can occur in the presence of a crack [4].

Generally speaking, it is not possible to draw a sharp line between brittle and ductile fracture, as some plastic deformation occurs in all types of fracture. This suggests that there is no such thing as a pure brittle fracture and that this type of fracture could be more accurately described as a quasi-brittle fracture.

Ductile fracture is characterized by plastic deformation in all fracture phases and occurs at a stress that is significantly higher than the yield stress. For a ductile fracture to occur, it is not necessary for a crack to form and propagate. Ductile fractures are transcrystalline because the crack grows through the crystalline grains [5–7].

Brittle fractures are believed to occur when the maximum tensile stress at a point reaches a critical value. The occurrence of brittle fractures is related both to the structural composition of the metal and to the operating conditions, in particular the working temperature, load rate

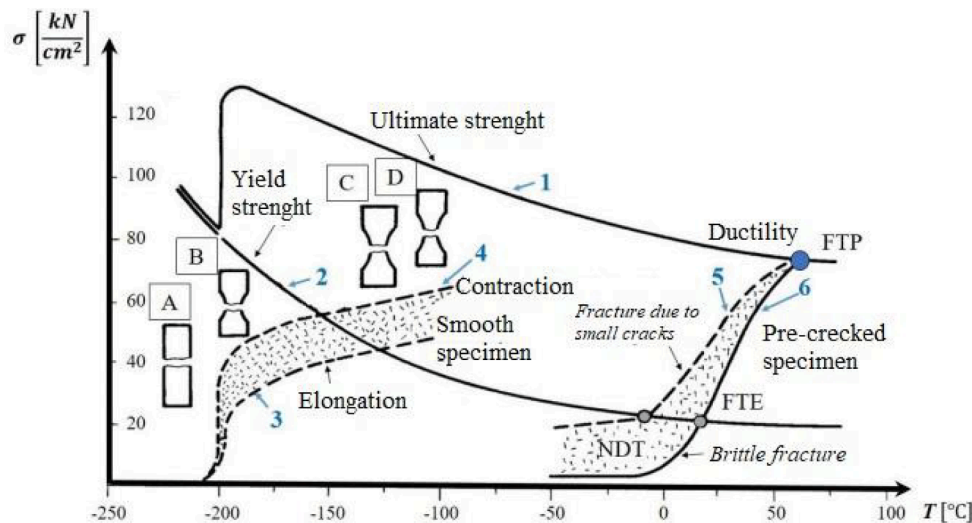


Fig. 3. Change in the mechanical properties of steel with temperature change [4].

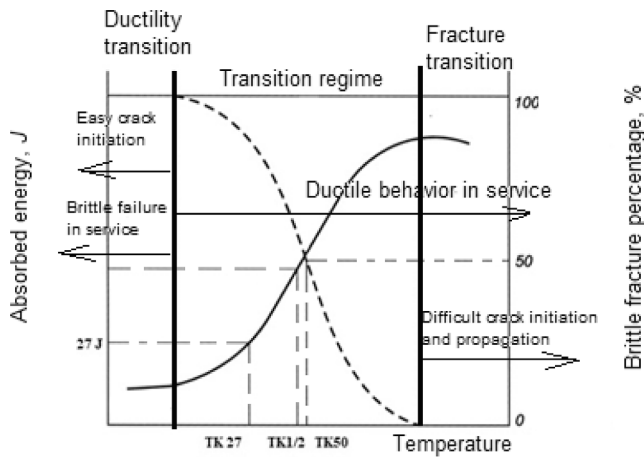


Fig. 4. Effect of temperature on absorbed impact energy and different transition temperatures [19].

and the presence of a notch. From a technical point of view, brittle fracture is a fracture that occurs with minimal plastic deformation and is conditionally referred to as brittle fracture.

Factors that can influence BDTT include temperature decrease, strain rate and the presence of stress concentrators, [8,9]. In the present work, particular attention was paid to the effect of temperature on the absorbed fracture energy in a temperature interval of  $-60\text{ }^{\circ}\text{C}$  to  $+40\text{ }^{\circ}\text{C}$ .

For practical reasons, the Charpy impact test is widely used as a quality control instrument in industry, since it enables simple evaluation of material behavior in presence of notches and cracks, [10,11]. Charpy impact tests clearly show a change in failure mode associated with a change in absorbed energy, from brittle cleavage fracture and a lower shelf energy at low temperatures to ductile cleavage fracture and an upper shelf energy at higher temperatures. The transition temperature is defined as the temperature range in which the change in energy values from the lower to the upper shelf occurs, [8,12–14].

Various Charpy test specimens are defined in the standard, [15]. The most commonly used are Charpy V specimens (V-notch, notch radius 0.25 mm, notch depth 2 mm). Other specimens, such as Charpy U specimens (U-notch, notch radius mm, notch depth 5 mm), are also used in the standard. As the notch depth increases, the transition temperature also shifts to higher values.

There are at least three different definitions of NDT, as follows and as shown in Fig. 4, [16–18]:

Table 1  
Chemical composition of NIOMOL 490 K, wt. %.

C	Si	Mn	P	S	Cr	Cu	Al
0.09	0.34	1.06	0.009	0.002	0.12	0.17	0.41
Sn	Ni	Mo	As	Nb	N	O	*C <sub>eq</sub>
0.005	0.9	0.26	0.008	0.06	0.0082	0.0075	0.414

$$*C_{eq} = C + \frac{Mn}{6} + \left(\frac{Cr + Mo + V}{5}\right) + \frac{(Ni + Cu)}{15}$$

- Temperature at a conventional level of Charpy energy (generally 27 J),  $T_{K27}$ .
- Temperature at half the jump between brittle and ductile plateau,  $T_{K1/2}$ , [16].
- Temperature corresponding to 50 % of fracture crystallinity  $T_{K50}$  [18], determined based on the appearance of the fracture surface and represents the temperature at which an equal share of ductile and brittle fracture occurs.

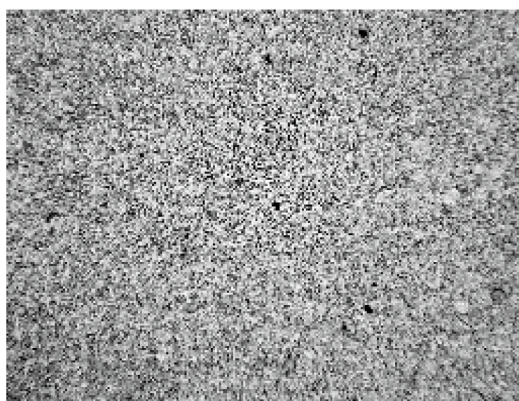
In this research, the first definition is used, as the most common and the simplest to evaluate. Standard Charpy impact test was used to evaluate NDT of all three zones of the microalloyed structural steel NIOMOL 490 K, parent metal (PM), weld metal (WM) and heat-affected-zone (HAZ).

## 2. Material and methods

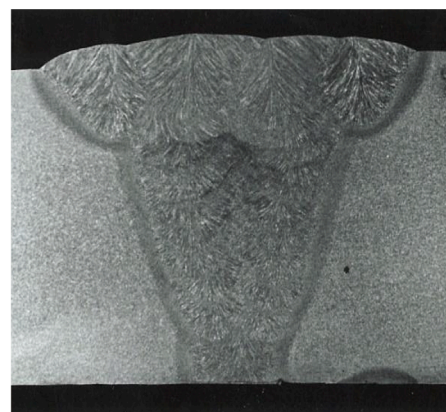
### 2.1. Material

NIOMOL 490 K steel belongs to the second generation of molybdenum-containing microalloyed steels with a minimum yield strength of 490 MPa, which has a guaranteed brittle fracture transition temperature of  $-60\text{ }^{\circ}\text{C}$ . It is primarily intended for the manufacture of pressure vessels in the petrochemical industry. NIOMOL 490 K is a thermomechanical hot-rolled steel with controlled cooling and a fine-grained microstructure that has good mechanical properties, good toughness even at low temperatures and a low carbon equivalent  $C_{ekv}$ . The bainitic-ferritic microstructure shown in Fig. 5(a) offers good resistance to hydrogen embrittlement, making this material suitable for the manufacture of welded structures that come into contact with aggressive environments with active hydrogen. The successful use of this steel depends on the extent to which the properties of the parent steel deteriorate during welding, [20–22].

The chemical composition and mechanical properties (yield strength – YS, tensile strength – TS, total elongation – A and impact energy according to ISO – V) tested at room temperature (RT) of parent material



(a)

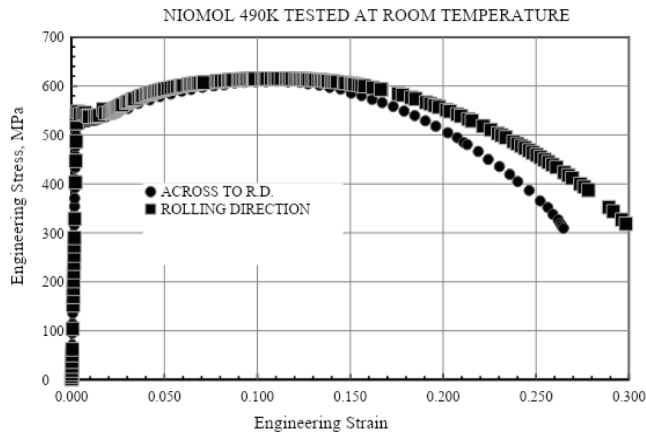


(b)

Fig. 5. Microstructure (a) parent steel (x 200, 3% Nital etching), (b) weld metal cross-section.

**Table 2**  
Mechanical properties of the investigated steel.

Direction	YS, MPa	TS, MPa	A, %	ISO-V, J
L-T	560	615	20.8	260
T-L	520	610	26.4	252



**Fig. 6.** Engineering stress–strain diagram for PM tested at RT in the rolling direction (L-T) and perpendicular to the rolling direction (T-L).

are given in [Table 1](#) and [Table 2](#), respectively. The value of the equivalent carbon content ( $C_{eq}$ ) is determined according to the formula given by the International Institute of Welding.

In order to take into account the influence of the rolling directions on the mechanical behavior of the tested welded plates, the mechanical properties were determined on specimens cut from the plate in the rolling direction (L-T) and perpendicular to the rolling direction (T-L). Engineering stress–strain curves for PM tested at RT, which were determined in the L-T and T-L directions, are shown in [Fig. 6](#).

## 2.2. Welding technology

The heat-affected zone (HAZ) and the weld metal (WM) are locations of reduced toughness where the brittle transition temperature is shifted to higher temperatures. Charpy V specimen testing, in which the notch tip is positioned in the WM and in various areas of the HAZ, is a recognized method for determining the impact strength of welded joints.

The scatter of impact toughness results is a consequence of the heterogeneity of the microstructure as well as the mechanical properties of the zones where the notch tip is located, and the width of the HAZ depends on the parameters of the welding process. The scatter may also be a consequence of the large transition radius of the V-shaped weld, which causes a stress concentration compared to the overall size of the HAZ with lower remote stresses.

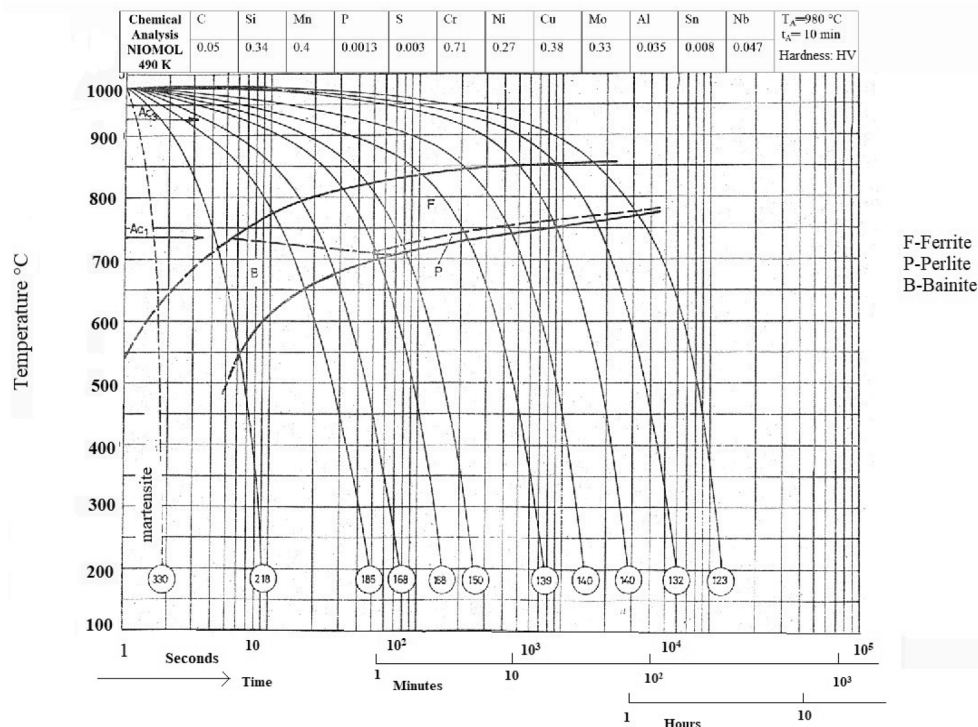
The time–temperature transformation diagram (TTT diagram) is shown in [Fig. 7](#), indicating the kinetics of isothermal transformations for steel of a given chemical composition austenitized at a temperature of  $t_A = 980\text{ }^\circ\text{C}$ .

The TTT diagram shows that NIOMOL 490 K is very similar to the boiler steel, which means that it is actually a boiler steel with twice the yield strength and higher toughness. It is practically impossible to cool this steel so quickly that martensite forms in its microstructure. Preheating during welding is not necessarily due to the low  $C_{eq}$ . [\[23\]](#).

The welding regime of the investigated steel NIOMOL 490 K requires the selection of proper parameters to avoid deterioration of the mechanical properties, [\[24\]](#). The thermal welding cycle depends on the heat input, the material thickness, parent material (PM) operating temperature and preheating temperature, as well as the shape and size of the welded joint.

Considering the fact that the steel investigated in this work is to be used for the manufacture of welded pressure vessels, submerged arc welding (SAW) was chosen as a highly productive and automated process for welding plates.

Two 30 mm thick plates with longitudinal butt welds were welded in horizontal position using SAW process using flux OP 40 TT. The filler metals used for welding the tested steel was  $\varnothing 3.25\text{ mm}$  SAW wire



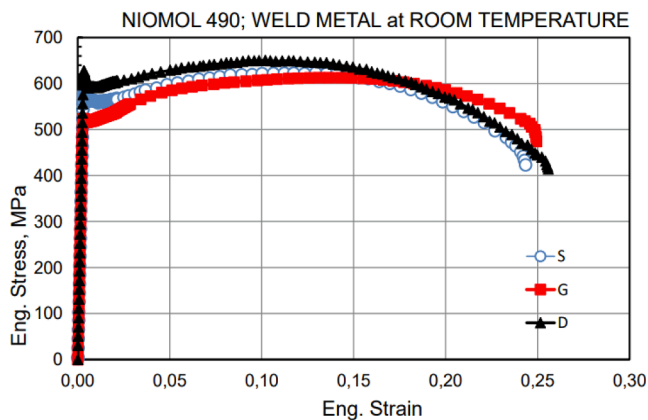
**Fig. 7.** Time temperature transformation diagram for NIOMOL 490 K steel [\[23\]](#).

**Table 3**  
Chemical composition of the filler metal.

Filler Metal	Elements %									
	C	Si	Mn	Cr	Mo	Ni	S	P	Cu	Ti
SAW NiMO <sub>2</sub>	0.08	0.28	1.05	0.05	0.36	1.6	0.008	0.022	0.19	–

**Table 4**  
Tensile properties of the weld metal (mean values) at RT.

Position	YS, MPa	TS, MPa	A, %
Top of the weld	516	612	24.7
Root of the weld	592	649	25.5
Center of the weld	543	646	24.3



**Fig. 8.** Stress–strain diagrams for three positions in WM (G-top; S-center and D-root).

NiMo<sub>2</sub>. The specified chemical composition of the filler metals is given in Table 3. The cross-section of a V-shaped weld seam is shown in Fig. 5 (b).

2.3. Tensile testing

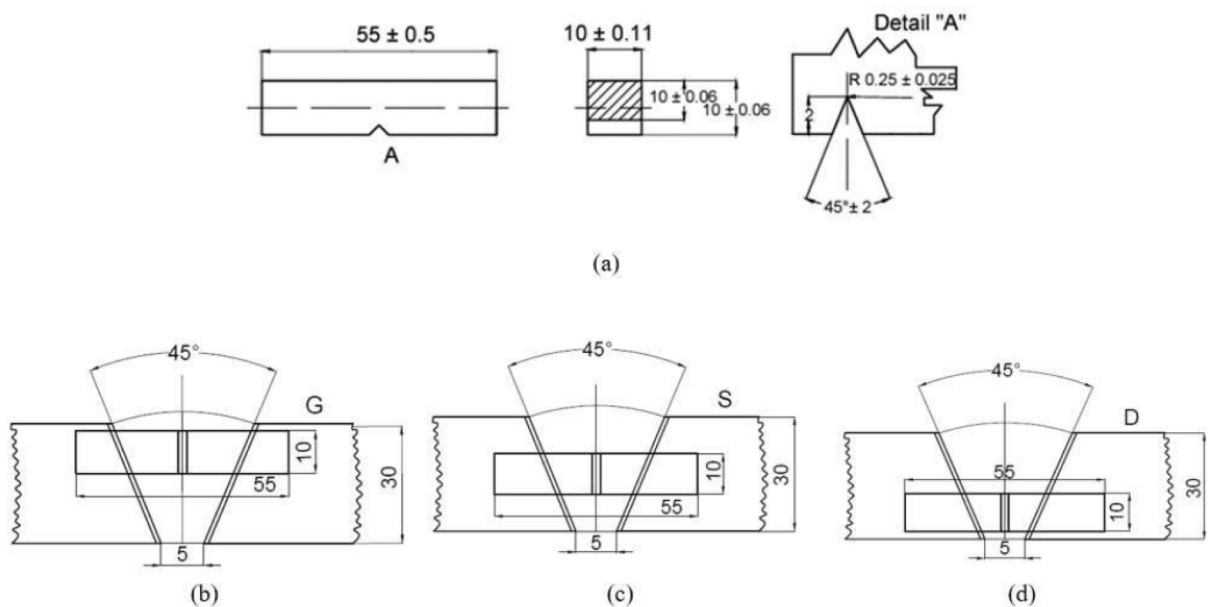
Tensile properties were determined on three types of specimens taken from the WM: from the face of the weld (top of the weld) labeled “G”, from the center of the weld labeled “S” and from the root of the weld labeled “D”. The average values of the tensile strength properties of WM are shown in Table 4 and the corresponding diagrams in Fig. 8.

2.4. Charpy impact testing

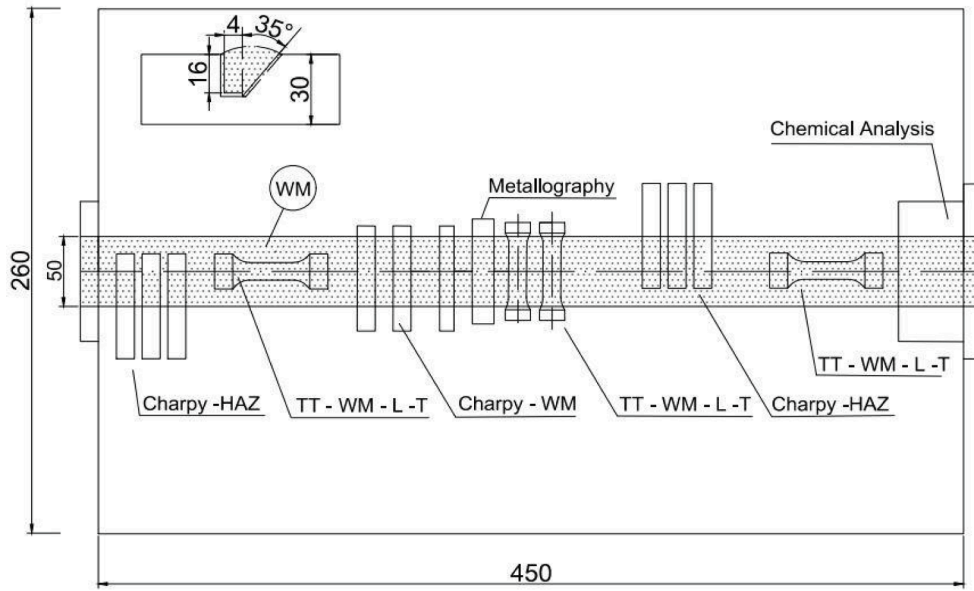
In accordance with the recommendations for standard notched impact testing of metallic ferrous materials, ASTM E23-2023, Charpy V-notch specimens were prepared (10 x 10 x 55 mm bars with a central V-notch of 2 mm depth and 0.25 mm notch radius), Fig. 9 (a), [25]. Three sets of Charpy-V specimens were made across the thickness of the welded plate: with a notch at the top in Fig. 9(b), at the center in Fig. 9(c) and at the root in Fig. 9(d) of the weld metal. The tests were carried out on an instrumented AMSLER RPK 150/300 J Charpy pendulum with a rigid C-pendulum. The Charpy notched bar impact test shows the correlation of the transition from ductile to brittle energy over a range of test temperatures. The amount of dissipated fracture energy depends on the fracture toughness of the steel as a function of temperature.

The sampling scheme for Charpy specimens from a 30 mm thick welded plate is shown in Fig. 10. From the welded plate with a weld width of 50 mm, specimens were cut for the analysis of the chemical composition of the virgin material and the WM, metallographic specimens for microstructural analysis, round tensile specimens in two rolling directions (L-T and T-L), and Charpy-V specimens perpendicular to the weld direction, Fig. 10(a). Fig. 10(b) shows the specimen sampling scheme with the cross-sectional views of the V-shaped weld showing the position of the Charpy notch in the HAZ.

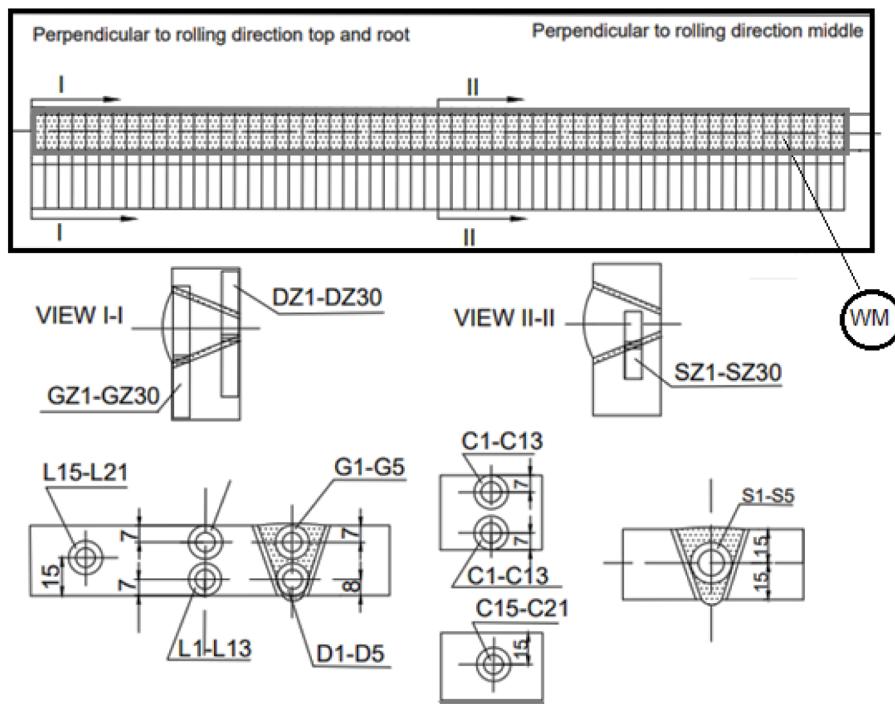
Three types of specimens from PM, WM, and HAZ were tested in a temperature range from –60 °C to 60 °C. Following the



**Fig. 9.** (a) Charpy V-notch impact test specimen geometry and specimens cut from the welded plate with the V-notch at (b) the top, (c) in the center and (d) at the root of WM.



(a)



(b)

Fig. 10. Sampling scheme of the Charpy specimens from the NIOMOL 490 K welded plate.

recommendations in [15] once the specimens reached the specified test temperature, they were annealed at the required temperature for 10 min and then quickly positioned and centered on the anvils and tested in less than 10 s.

### 3. Results and discussion

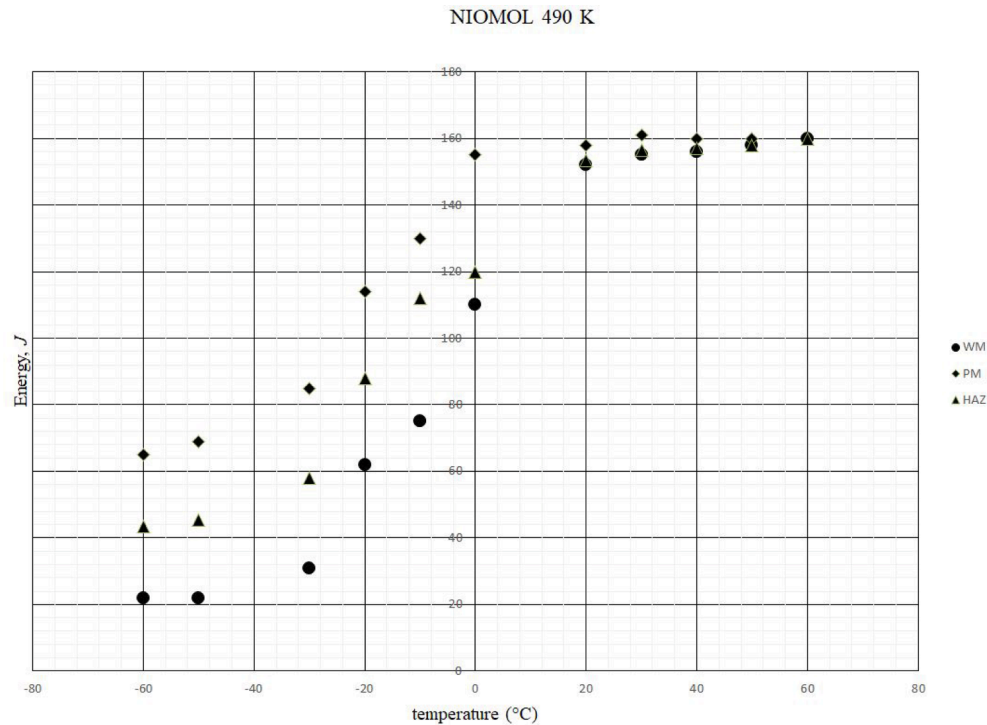
Values of the total impact energy at all test temperatures are given in Table 5 and Fig. 11 for three zones of NIOMOL 490 K welded joint. The NDT is obtained from Fig. 11, using the criterion  $T_{K27}$ . The NDT values

for different welded joint constituents of NIOMOL 490 K are shown in Table 6.

It should be noted that both PM and HAZ have a NDT below  $-60\text{ }^{\circ}\text{C}$ , while WM shows a more sensitive behavior in this respect, as the NDT is at  $-40\text{ }^{\circ}\text{C}$ , which limits the use of NIOMOL 490 K welded structures. Of course, if lower operating temperatures are required, other welding processes and/or parameters must be used. Based on the data in [26,27], the use of the GSAW process with a gas mixture (5 %  $\text{CO}_2$ , 1 %  $\text{O}_2$ , balance Ar) can be recommended to obtain acicular microstructure in WM, that is less sensitive to the presence of notches and cracks than

**Table 5**  
Impact test results of NIOMOL 490 K.

Specimen	PM								
temperature, °C	-60	-50	-30	-20	-10	0	20	30	40
$E_{tot}, J$	65	69	85	114	130	155	158	161	161
Specimen	WM								
temperature, °C	-60	-50	-30	-20	-10	0	20	30	40
$E_{tot}, J$	22	22	31	62	75	110	152	155	156
Specimen	HAZ								
temperature, °C	-60	-50	-30	-20	-10	0	20	30	40
$E_{tot}, J$	44	46	58	88	112	120	154	157	157



**Fig. 11.** Charpy notched impact transition temperatures for PM, WM and HAZ of NIOMOL 490 K.

**Table 6**  
NDT temperatures of the welded joint made of NIOMOL 490 K.

Material	PM	WM	HAZ
NDT	Below -60 °C	Below -60 °C	-40 °C

SAW. It can be concluded that the more productive process is not the best option in this case. It can be said that the results obtained are as expected for a micro-alloyed fine grain steel such as NIOMOL 490 K, as the SAW process usually produces WM with a less fine grain.

Finally, it should be noted that the second criterion for the evaluation of the NDT temperature (half of the jump between the brittle and ductile plateau,  $TK_{1/2}$ , [16]) is not suitable in this case, since its estimates would be unrealistically high for all welded joint zones, around 0 °C, with corresponding energies around 100 J, which certainly does not represent brittle behavior.

**4. Conclusions**

This article deals with the NDT temperature of welded joints made by SAW, which were determined using the Charpy impact test. Based on the results presented, the following can be concluded:

- Welded joints made of NIOMOL 490 K steel produced using the SAW process are not suitable for use at temperatures below -40 °C, as the WM impact toughness is below 27 J. Instead, the use of the GSAW process with a gas mixture (5 % CO<sub>2</sub>, 1 % O<sub>2</sub>, remainder Ar) is recommended.
- -The second criterion for evaluating the NDT temperature, which is based on half of the jump between the brittle and ductile plateau, is not suitable in the case investigated here.

**CRedit authorship contribution statement**

**Ana Maksimović:** Methodology, Investigation, Conceptualization, Project administration, Resources, Writing – original draft. **Ljubica Milović:** Supervision, Visualization, Writing – review & editing. **Bojana Zečević:** Supervision. **Vujadin Aleksić:** Data curation, Formal analysis. **Dragoljub Bekrić:** Conceptualization.

**Declaration of competing interest**

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Ana Maksimovic reports administrative support was provided by Republic of Serbia Ministry of Education Science and Technological Development.

## Data availability

Data will be made available on request.

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