# Primerjava različnih metod preizkušanja jekla NIOMOL 490 za določevanje lomnih karakteristik pri nizkih temperaturah

# Comparison of different tests on NIOMOL 490 steel to determine fracture characteristics at low temperature

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**ASM/SLA: Q26s, Q6n, SGBr, 1—54**

*V prispevku so obravnavane lomne značilnosti drobnozrnatega mikrolegiranega jekla NIOMOL 490. To jeklo ima feritno bainitno mikrostrukturo ter mejo plastičnosti minimalno 490 MPa, dobro duktilnost pa ima še tudi pri temperaturi −60° C. Staranje tega jekla pa duktilne lastnosti poslabša, zato nevarnost krhkega loma postane realna. Lastnosti tega jekla v odvisnosti od temperature uporabe so bile določene tako s statičnimi preizkusi, kot tudi z udarnimi preizkusi ter merjenjem lomne žilavosti.*

## 1. UVOD

Nosilni element konstrukcije odpove zaradi prekoračitve mejne napetosti, zaradi nestabilnosti ali pa zaradi loma. Prva dva kriterija sta v konstruktorski praksi že dolgo znana, njuna uporaba pa je predpisana s standardi. V novejšem času, ko se zaradi tehnoloških zahtev uvajajo materiali izredno visokih trdnosti, pa prva dva kriterija za varnost nosilnega elementa nista več zadostna. Zasnovana sta na predpostavki, da je material homogen in izotropen, ter ne upoštevata napak v materialu, ki med eksploatacijo lahko prerastejo v razpoke kritične velikosti, ki povzročijo porušitev zaradi loma.

Pojav krhkega loma lahko pričakujemo tudi pri jeklu NIOMOL 490, ki spada v skupino mikrolegiranih drobnozrnatih jekel s feritno bainitno mikrostrukturo ter mejo plastičnosti min 490 MPa. Jeklo je zaradi dobre duktilnosti še tudi pri temperaturi −60° C primerno za izdelavo tlačnih posod za utekočinjene pline. Ravno pri teh konstrukcijah pa se nevarnost pojavljanja krhkega loma v praksi še stopnjuje zaradi procesov staranja predhodno v hladnem deformiranega jekla (podnice).

Zato moramo tlačne posode kontrolirati tudi s stališča lomne mehanike, ki se ukvarja z vplivom atomarno ostre razpoke v nosilnem materialu. Ob tem pa moramo poznati lomne značilnosti jekla, da lahko določimo temperaturno mejo uporabnosti tega jekla.

## 2. VRSTA PREISKAV

Za določanje lomnih značilnosti drobnozrnatega mikrolegiranega jekla NIOMOL 490 smo uporabili statične in dinamične mehanske preizkuse. Iz skupine statičnih

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*The article presents fracture characteristics of the fine grained microalloyed NIOMOL steel. This steel's microstructure is ferritic-bainitic and its minimal yield strength 490 MPa. It is good at ductility even at −60° C. Aging impairs the ductile properties, causing brittle fracture. The steel properties in relation to working temperatures have been determined by static load testing as well as impact testing, and measurements of fracture toughness.*

## *1. INTRODUCTION*

*Exceded stress threshold, instability or fracture cause a construction bearing element to fail. For a long time the first two criteria have been known to builders and their application specified by standards. Recently, when high strength materials were required by development of tehnology, the two first mentioned criteria became insufficient because the supposition of homogenous and isotropic material did not consider flaws in the material which could develop cracks of critical size causing colapse.*

*NIOMOL 490, belonging to the group of fine grained microalloyed steels with a ferritic bainitic microstructure and a minumum yield stress of 490 MPa might become subject to brittle fracture. The steel shows good ductility at −60° C, thus it is suitable for high pressure vessels for liquid gases. Such constructions are endangered by brittle fracture because of the aging of the cold formed steel bottom. Therefore, the high pressure vessels have to be checked for fractures in the bearing material. For this reason it is neccessary to know the fracture characteristics of the steel, and to define the temperature boundary for the use of this steel.*

## *2. TEST TYPES*

*To determine fracture characteristics, static load and dynamic mechanical tests were performed. The conventional tensile test, the tensile test of specimens with a circumferential notch and measurement of fracture toughness with the J integral method and correction by Schwalbe, were chosen from the group of static mechanical tests.*

*The impact Charpy-V method for toughness measurement and the method for the nill ductility temperature determination by drop weight test was selected from the group of dynamic mechanical tests.*

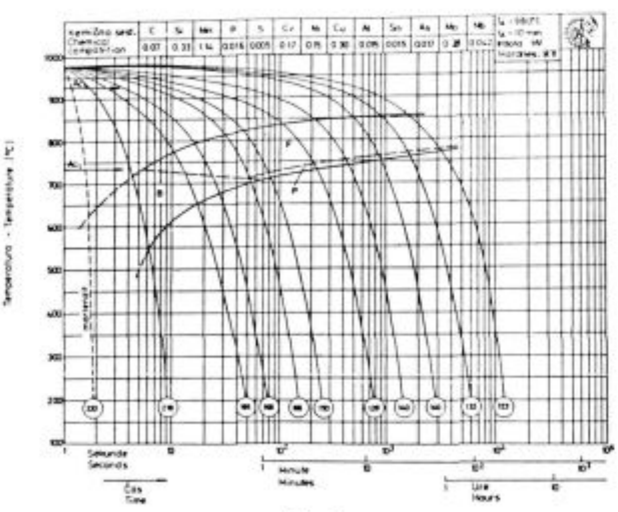
mehanskih testov smo izbrali konvencionalni natezni preizkus, natezni preizkus cilindričnih preizkušancev z obodno zarezo ter merjenje lomne žilavosti z metodo J integrala ter korekcijo po Schwalbeju. Iz skupine dinamičnih mehanskih preizkusov pa smo izbrali metodo udarnega merjenja žilavosti Charpy-V in metodo določanja temperature neduktilnega loma (drop weight test).

## 3. EKSPERIMENTALNI DEL

### 3.1 Določanje trdnostnih karakteristik jekla NIOMOL 490 in NIOMOL 490 K

Za preiskave smo uporabili drobnozrnato mikrolegirano jeklo NIOMOL 490, debeline 12 mm, z mejo plastičnosti 490 MPa. To jeklo spada med mikrolegirana jekla, legirana z Mn, Mo, Nb s feritno bainitno mikrostrukturo. Železarna Jesenice ga je dobavila v normaliziranem stanju. Mehanske lastnosti in kemična analiza so razvidne iz **tabel I, II, III**. Izoblikovanje mikrostrukture jekla NIOMOL 490 je prikazano s TTT diagramom kontinuirnega ohlajanja na **sliki 1**. Mikrostruktura jekla NIOMOL 490 je prikazana na **sliki 2**. Sestavljata jo ferit in bainit. Iz **slike 2** je razvidno, da je jeklo izrazito drobnozrnato in ima feritno-bainitno mikrostrukturo z dokajšnjim deležem bainita. Delež bainita v mikrostrukturi pa domnevno zaradi segregiranja, zlasti ogljika, nekoliko variira.

Del preiskav smo opravili tudi na jeklu NIOMOL 490 K, zato prikazujemo tudi njegove karakteristike — **tabela IV, V, VI**.



**Slika 1.**
TTT diagram jekla NIOMOL 490
***Figure 1.***
*TTT diagram of the NIOMOL 490 steel*

**Tabela 1:** Mehanske lastnosti pločevine Niomol 490

| Pločevina Niomol 490 t = 12 mm | Mehanske lastnosti pločevine | | | | |
|---|---|---|---|---|---|
| | Rp MPa | Rm MPa | A5 % | Z % | Smer preizkušanja |
| Potrdilo o kvaliteti Železarne Jesenice št. 11759 | 488 | 614 | 26 | — | prečno na smer valjanja |
| Podatki iz prospekta Železarne Jesenice | 490 | 560 – – 740 | 19 | — | |

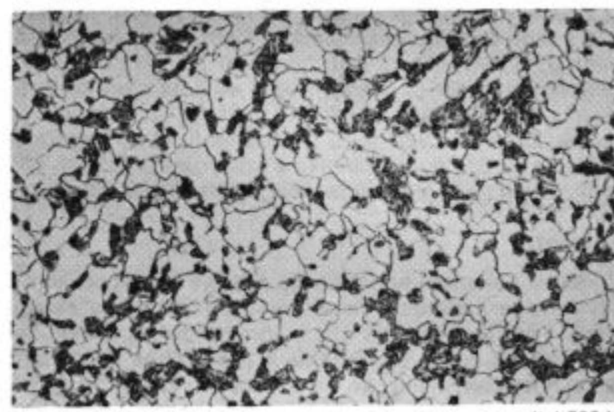
## *3.0 EXPERIMENTS*

### *3.1 Tensile strength characteristic*

*All specimens were manufactured from a 12 mm thick plate with yield strength 490 MPa and delivered after normalising by Jesenice steelworks. The NIOMOL 490 and 490 K steels belong to microalloyed steels alloyed with Mn, Mo, Nb with a ferritic-bainitic microstructure. Their mechanical properties and the chemical composition are shown in* ***table I., II. and III.*** *The TTT diagram for continuous cooling of the NIOMOL 490 K steel is shown in* ***Figure 1.***

*The microstructure of the steel shown in Figure 2 consists of ferrite and bainite and is finegrained. The share of bainite in the microstructure varries to a certain extent because of local seggregations, mostly carbonous.*

*The investigation was partly carried out on the NIOMOL 490 K steel, whose characteristics are shown in*



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**Slika 2.**
Mikrostruktura jekla NIOMOL;490
***Figure 2.***
*Microstructure of the NIOMOL 490*

**Tabela 2:** Kemijska sestava pločevine Niomol 490

| Kemijska sestava pločevine Niomol 490 t = 12 mm | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Oznaka | C | Si | Mn | P | S | Cr | Ni | Cu | Mo | Al | Nb |
| | % | | | | | | | | | | |
| 1 | 0.11 | 0.26 | 1.20 | 0.018 | 0.006 | 0.24 | 0.30 | 0.18 | 0.26 | 0.031 | 0.054 |
| 2 | 0.09 | 0.24 | 1.21 | 0.018 | 0.006 | — | — | — | — | — | — |

1. Dejanska analiza pločevine
2. Podatki iz atesta Železarne Jesenice št. 11759

**Tabela 3:** Žilavost pločevine Niomol 490

| Smer valjanja | Žilavost ISO — V (J) nestarano stanje | | | | | | Žilavost DVM (J) starano stanje | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Temperatura preizkušanja | | | | | | | | | | |
| | +20 | 0 | −20 | −40 | −50 | −60 | +20 | + 5 | −20 | −40 | −60 |
| Vzdolžno | 63 | 63 | 63 | 55 | 47 | 39 | 47 | 41 | 41 | 31 | 27 |
| Prečno | 55 | 55 | 47 | 39 | 34 | 31 | 35 | 31 | 31 | 27 | — |

**Tabela 4:** Mehanske lastnosti pločevine Niomol 490 K

| Mehanske lastnosti pločevine Niomol 490 K t = 24 mm | | | | | |
|---|---|---|---|---|---|
| | Rp MPa | Rm MPa | A5 % | Z % | Smer preizkušanja |
| Potrdilo o kvaliteti Železarne Jesenice | 496<br>529 | 564<br>604 | 21.8<br>19.8 | —<br>— | prečno na smer valjanja |

**Tabela 5:** Kemijska sestava pločevine Niomol 490 K

| Kemijska sestava pločevine Niomol 490 K t = 24 mm | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C | Si | Mn | P | S | Cr | Ni | Cu | Mo | Al | Nb | Ti | N |
| % | | | | | | | | | | | | |
| 0.09 | 0.31 | 0.33 | 0.012 | 0.002 | 0.53 | 0.18 | 0.37 | 0.28 | 0.052 | 0.052 | 0.022 | 0.0093 |

**Tabela 6:** Žilavost pločevine Niomol 490 K

| Smer valjanja | Žilavost ISO — V (J) nestarano stanje | | | Žilavost ISO — V (J) nestarano stanje | | |
|---|---|---|---|---|---|---|
| | Temperatura preizkušanja (°C) | | | | | |
| | −20 | | | −60 | | |
| Prečno | 300<br>228 | 249<br>238 | 300<br>240 | 162<br>163 | 166<br>161 | 163<br>160 |

Mikrostruktura jekla NIOMOL 490 K je razvidna s **slike 3**. Mikrostruktura je podobna kot na **sliki** 2, vendar je delež bainita nižji, medtem ko so kristalna zrna ferita približno enako velika, kot pri jeklu NIOMOL 490.

Iz jekla NIOMOL 490 smo izdelali različne vrste preizkušancev in določili mehanske lastnosti te pločevine, ki so razvidne iz **tabele VII**. Natezni preizkus cilindričnih preizkušancev z obodno zarezo iz jekla NIOMOL 490 v dobavnem stanju, kot tudi 10 % deformiranem v hladnem ter umetno staranem 30'/250° C je razviden iz **tabele VIII in IX**.

Na osnovi zbranih rezultatov je razvidno, da jeklo NIOMOL 490 v pogojih statičnega preizkušanja ne kaže bistvenega poslabšanja lastnosti vse do temperature −100° C. Lastnosti tega jekla v dobavnem stanju so si-

***Table 1:** Mechanical properties of Niomol 490 in 12 mm plate*

| *Plates Niomol 490 t = 12 mm* | *Mechanical Properties* | | | | |
|---|---|---|---|---|---|
| | *Rp MPa* | *Rm MPa* | *A5 %* | *Z %* | *Direction of Testing* |
| Quality certificate issued by Jesenice Steelworks No. 11759 | 488 | 614 | 26 | — | transverse |
| Data from Jesenice Steelworks booklet | 490 | 560 − − 740 | 19 | — | |

***Table 2:** Chemical composition of Niomol 490 steel*

| *Chemical Composition of Niomol 490 Plates t = 12 mm* | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Designation | C | Si | Mn | P | S | Cr | Ni | Cu | Mo | Al | Nb |
| | % | | | | | | | | | | |
| 1 | 0.11 | 0.26 | 1.20 | 0.018 | 0.006 | 0.24 | 0.30 | 0.18 | 0.26 | 0.031 | 0.054 |
| 2 | 0.09 | 0.24 | 1.21 | 0.018 | 0.006 | — | — | — | — | — | — |

*1. Analysis of the tested plate*
*2. Data from the Jesenice Steelworks certificate No. 11759*

***Table 3:** Impact toughness of Niomol 490*

| *Rolling direction* | *Impact Toughness ISO-V (J) not Aged* | | | | | | *Impact Toughness DVM (J) Aged* | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Test temperature* | | | | | | | | | | |
| | +20 | 0 | −20 | −40 | −50 | −60 | +20 | + 5 | −20 | −40 | −60 |
| Longitudinal | 63 | 63 | 63 | 55 | 47 | 39 | 47 | 41 | 41 | 31 | 27 |
| Transversal | 55 | 55 | 47 | 39 | 34 | 31 | 35 | 31 | 31 | 27 | — |

***Table 4:** Mechanical properties of Niomol 490 K steel*

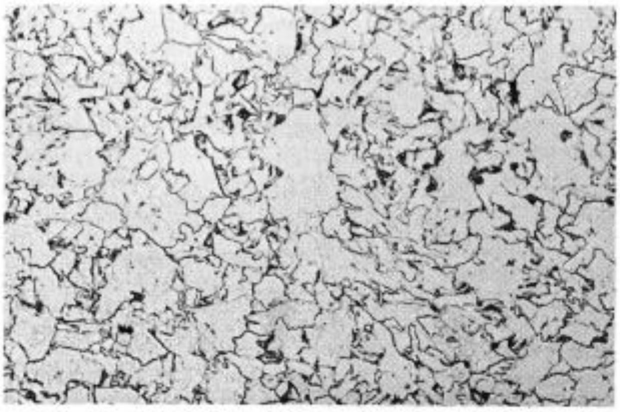
| *Mechanical properties of Niomol 490 K plates t = 24 mm* | | | | | |
|---|---|---|---|---|---|
| | *Rp MPa* | *Rm MPa* | *A5 %* | *Z %* | *Direction of Testing* |
| Quality certificate issued by Steelworks Jesenice | 496<br>529 | 564<br>604 | 21.8<br>19.8 | —<br>— | Transverse |

***Table 5:** Chemical composition of the Niomol 490 K steel*

| *Chemical composition of Niomol 490 K plates t = 24 mm* | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| *C* | *Si* | *Mn* | *P* | *S* | *Cr* | *Ni* | *Cu* | *Mo* | *Al* | *Nb* | *Ti* |
| % | | | | | | | | | | | |
| 0.09 | 0.31 | 0.33 | 0.012 | 0.002 | 0.53 | 0.18 | 0.37 | 0.28 | 0.052 | 0.052 | 0.022 |

***Table 6:** Impact toughness of the Niomol 490 K steel*

| *Rolling direction* | *Impact toughness ISO-V (J) not aged* | | | *Impact toughness ISO-V (J) not aged* | | |
|---|---|---|---|---|---|---|
| | *Test temperature (°C)* | | | | | |
| | −20 | | | −60 | | |
| Transversal | 300<br>228 | 249<br>238 | 300<br>240 | 162<br>163 | 166<br>161 | 163<br>160 |



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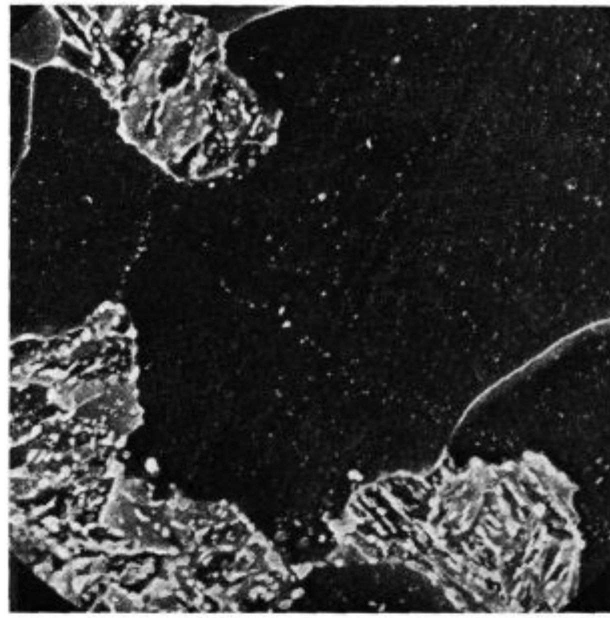

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**Slika 3.**
Mikrostruktura jekla NIOMOL 490 K

***Figure 3.***
*Microstructure of the NIOMOL 490 K*

cer znatno boljše od lastnosti deformiranega in staranega jekla, vendar pa je primerjava teh lastnosti v odvisnosti od temperature preizkušanja praktično enaka. Izkaže se tudi, da je enakomerni raztezek boljši kazalnik duktilnosti jekla, kot pa je to zarezno trdnostno razmerje, kar je pravzaprav presenetljivo, če upoštevamo, da enakomerni raztezek meri le največjo dosegljivo homogeno deformacijo, z zareznim trdnostnim razmerjem pa merimo tudi sposobnost utrjevanja jekla ob zarezi. Podobno pa velja tudi za lomno duktilnost, ki je dobra mera za de-

***tables IV., V. and VI.*** *The microstructure of the NIOMOL 490 K steel is shown in* ***Figure 3.*** *The grain size is similar to that in* ***Figure 2,*** *while the amount of bainite is smaller.*

*The mechanical properties of 490 steel are shown in* ***table VII.*** *The tensile test results of cylindrical specimens with a circumferential notch made from the NIOMOL 490 steel in the as delivered condition as well as after 10 % cold deformation and aging for 30 min at 250° C are shown in* ***table VIII. and IX.***

**Tabela 7:** Mehanske lastnosti drobnozrnatega mikrolegiranega jekla Niomol 490 t = 12 mm
***Table 7:*** *Mechanical properties of the finegrained microalloyed steel Niomol 490 t = 12 mm*

| *Vrsta preizkušanca in trgalni stroj* | *Oznaka epruvete* | *Odvzem preizkušanca vzdolž smeri valjanja pločevine* | | | | | *Oznaka epruvete* | *Odvzem preikušanca pravokotno na smer valjanja pločevine* | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | *Rp* | *Rm* | $A_5$ | $e_{gt}$ | *Z* | | *Rp* | *Rm* | $A_5$ | $e_{gt}$ | *Z* |
| | | *Mpa* | | | *%* | | | *Mpa* | | | *%* | |
| »minitrac« ∅ 5 mm, Amsler 100 kN | 2,1 | 482 | 600 | 22,3 | 16,07 | 72 | 1,1 | 499 | 611 | 34,6 | 17,6 | 67,5 |
| | 2,2 | 477 | 599 | 23,1 | 17,0 | 72,7 | 1,2 | 484 | 593 | 35,8 | 17,9 | 71,9 |
| | 2,3 | 482 | 605 | 22,5 | 16,6 | 72 | 1,3 | 499 | 597 | 33,2 | 16,8 | 70,8 |
| Okrogla epruveta ∅ 10 mm/M 16 (strictiomax), Amsler 100 kN | 2,10 | 467 | 596 | 28,5 | 13,8 | 72,4 | 1,7 | 486 | 579 | 25,9 | 12,0 | 69,1 |
| | 2,11 | 461 | 586 | 28,5 | 14,2 | 72,9 | 1,8 | 476 | 584 | 18,8 | 12,2 | 70,1 |
| | 2,12 | 429 | 580 | 20,2 | 13,8 | 73 | 1,9 | 463 | 579 | 18,9 | 12,7 | 67,2 |
| Okrogla epruveta ∅ 10 mm, L = 250 mm, Instron 1343 | 2,15 | 458 | 586 | 25,2 | 12,70 | 71,9 | 1,13 | 467 | 586 | 24,7 | 13,40 | 68,6 |
| | 2,16 | 453 | 575 | 26,7 | 14,20 | 72,2 | 1,14 | 465 | 588 | 24,3 | 13,00 | 68,5 |
| | 2,17 | 458 | 586 | 25,2 | 12,70 | 71,9 | 1,15 | 483 | 579 | 25,2 | | 71,9 |
| »minitrac« ∅ 5 mm, odvzet s površine pločevine, Amsler 100 kN | 2,18 | 462 | 585 | 31 | | 78,8 | 1,16 | | | | | |
| | 2,19 | 465 | 583 | 28,4 | | 73,9 | 1,17 | | | | | |
| | 2,20 | 462 | 585 | 31 | | 78,8 | 1,18 | | | | | |
| Podatki s prospekta Železarne Jesenice | | | | | | | | 490 | 560 – 740 | 19 | | |

kjer pomeni:
$e_{gt}$ . . . max. enakomerni raztezek (%)

**Tabela 8:** Konvencionalni natezni preizkus in preizkus cilindričnih preizkušancev z obodno zarezo na jeklu Niomol 490 v dobavnem stanju

***Table 8:** Conventional tensile test and test on cylindrical specimens with circumferential notch of the steel Niomol 490 in as delivered condition*

| Osnovni material | Oblika epruvete | Oznaka epruvete | Temperatura preizkušanja | Osnovni premer epruvete (mm) | Podatki o preizkušancu: do / $d_k$ ($d_z$) (mm) | Podatki o preizkušancu: S / Su ($mm^2$) | lo (mm) | $l_k$ (mm) | $P_F$ (kN) | $P_m$ (kN) | Mehanske lastnosti: $R_p$ (MPa) | Mehanske lastnosti: $R_m$ (MPa) | Mehanske lastnosti: $e_{gr}$ (%) | $\varepsilon_u$ | $\varepsilon_l = \ln\frac{S_0}{S_u}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Niomol 490 t = 12 mm | brez zareze | 1,1 | T = -20°C | Ø 8 | 5,0<br>8,0 / 2,6<br>4,4 | 50,24<br>19.63 / 15,2<br>5,81 | 40<br>25 | 50,05<br>32,5 | 23,7<br>9,20 | 29,8<br>11,65 | 472<br>469 | 593<br>593 | 15,6<br>18,1 | 0,145<br>0,166 | 0,9397<br>0,967 |
| | brez zareze | 1,2 | T = -20°C | Ø 10 | 10,0 / 5,5 | 78,54 / 23,76 | 100 | 118,1 | 38,7 | 48 | 493 | 611 | 14,3 | 0,134 | 1,195 |
| | z zarezo | 1,3 | T = -20°C | Ø 8 | 3,78 / 2,90 | 11,22 / 6,60 | 25 | – | 7,44 | 8,98 | – | 800 | 2,1 | 0,021 | – |
| | z zarezo | 1,4 | T = -20°C | Ø 10 | 7,0 / 6,1 | 38,48 / 29,22 | 100 | – | 33,5 | 36,0 | – | 935 | 2,8 | 0,028 | – |
| | brez zareze | 1,5 | T = -40°C | Ø 8 | 8,0 / 4,6 | 50,27 / 16,62 | 40 | 49,4 | 30,1 | 37,5 | 599 | 746 | 16,9 | 0,156 | 1,106 |
| | brez zareze | 1,6 | T = -40°C | Ø 8 | 8,0 / 4,3 | 50,24 / 14,51 | 40 | 50,0 | 25,5 | 32,3 | 507 | 643 | 14,1 | 0,132 | 1,242 |
| | z zarezo | 1,7 | T = -40°C | Ø 8 | 5,6 / 5,1 | 24,63 / 20,43 | 40 | – | 25 | 28,4 | – | 1153 | 0,9 | 0,009 | – |
| | z zarezo | 1,8 | T = -40°C | Ø 8 | 5,5 / 4,4 | 23,75 / 15,20 | 40 | 40,5 | 19,24 | 23,6 | – | 994 | 1,4 | 0,014 | – |
| | brez zareze | 1,9 | T = -100°C | Ø 8 | 8,0 / 4,65 | 50,24 / 16,97 | 40 | 48,45 | 27,9 | 35,2 | 555 | 700 | 18,8 | 0,172 | 1,085 |
| | brez zareze | 1,10 | T = -100°C | Ø 8 | 8,0 / 4,4 | 50,24 / 15,20 | 40 | 50,0 | 27,2 | 33,8 | 542 | 673 | 16,3 | 0,151 | 1,195 |
| | z zarezo | 1,11 | T = -100°C | Ø 8 | 5,5<br>5,6 / 4,8 | 24,62<br>23,75 / 18,09 | 40<br>40 | 41,45 | 28,2<br>23,77 | 28,2<br>26,10 | – | 1146<br>1099 | 3,4 | 0,034 | – |
| | z zarezo | 1,12 | T = -100°C | Ø 8 | 5,5 / 4,7 | 23,75 / 17,34 | 40 | 41,25 | 24,51 | 26,9 | – | 1133 | 2,0 | 0,020 | – |
| Epruvete odvzete prečno na smer valjanja ≠ | brez zareze | 1,13 | T = -150°C | Ø 8 | 8,0 / 4,8 | 60,27 / 18,10 | 40 | 43,70 | 30,75 | 37,8 | 612 | 752 | 15,6 | 0,145 | 1,202 |
| | brez zareze | 1,14 | T = -150°C | Ø 8 | 8,0 / 5,1 | 50,24 / 20,42 | 40 | 48,45 | 35,3 | 41,7 | 702 | 830 | 15,6 | 0,145 | 0,900 |
| | z zarezo | 1,15 | T = -150°C | Ø 8 | 5,65 / – | 25,07 / – | 40 | – | 31,4 | 31,4 | – | 1252 | | | – |
| | z zarezo | 1,16 | T = -150°C | Ø 8 | 5,8 / 4,3 | 26,41 / 14,51 | 40 | 40,75 | 28,2 | 28,4 | – | 1113 | 1,7 | 0,016 | – |
| | brez zareze | 1,17 | T = -196°C | Ø 8 | 8,0 / 4,75 | 50,24 / 17,71 | 40 | 45,55 | 36,3 | 39,4 | 722 | 784 | 9,2 | 0,088 | 1,042 |
| | brez zareze | 1,18 | T = -196°C | Ø 8 | 8,0 / 4,9 | 50,24 / 18,85 | 40 | 47,3 | 32,3 | 39,3 | 644 | 782 | 8,7 | 0,084 | 0,980 |
| | z zarezo | 1,19 | T = -196°C | Ø 8 | 5,8 / 5,2 | 26,41 / 21,23 | 40 | 40,1 | 34,8 | 35,9 | – | 1359 | 0,4 | 0,004 | – |
| | z zarezo | 1,20 | T = -196°C | Ø 8 | 5,8 / 5,6 | 26,41 / 26,62 | 40 | 40,3 | 37,7 | 37,7 | – | 1427 | 0,4 | 0,004 | – |

**Tabela 9:** Konvencionalni natezni preizkus in preizkus cilindričnih preizkušancev z obodno zarezo na 10 % deformiranem in umetno staranem (250° C/30 minut) Niomolu 490

***Table 9:*** *Conventional tensile test and test on cylindrical specimens with circumferenial notch on 10 % strained and artifficaly aged (250° C/30 ') Niomol 490*

| Osnovni material | Oblika epruvete | Oznaka epruvete | Temperatura preizkušanja | Osnovni premer epruvete (mm) | Podatki o preizkušancu: do / $d_k$ ($d_l$) (mm) | S / Su ($mm^2$) | lo (mm) | $l_k$ (mm) | $P_p$ (kN) | $P_m$ (kN) | Mehanske lastnosti: $R_p$ (MPa) | $R_m$ (MPa) | $e_{gl}$ (%) | $\varepsilon_u$ | $\varepsilon_f = \ln \frac{S_0}{S_u}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Niomol 490 t = 12 mm 10 % hladno deformiran in staran 250°/30' | brez zareze | 2,1 | T = +20°C | Ø 8 | 8,0 / 5,01 | 50,27 / 19,71 | 40 | 46 | 33,58 | 34,40 | 668 | 684 | 2,75 | 0,0271 | 0,936 |
| | | | | | 4,7 / 3,0 | 17,35 / 7,07 | 25 | 28,5 | 12,3 | 13,72 | 624 | 696 | 1,4 | 0,014 | 0,897 |
| | brez zareze | 2,2 | T = +20°C | Ø 10 | 10,0 / 6,7 | 78,54 / 35,24 | 100 | 109,4 | 50,5 | 51,85 | 643 | 660 | 4,5 | 0,044 | 0,801 |
| | z zarezo | 2,3 | T = +20°C | Ø 8 | 3,52 / 3,10 | 9,73 / 7,55 | 25 | — | 10,70 | 10,70 | — | 1099 | 0,5 | 0,005 | — |
| | z zarezo | 2,4 | T = +20°C | Ø 10 | 6,83 / — | 36,64 / — | 100 | — | 39,75 | 39,75 | — | 921 | 3,3 | 0,032 | — |
| | brez zareze | 2,5 | T = -40°C | Ø 8 | 8,0 / 4,85 | 50,27 / 18,47 | 40 | 46,35 | 41,3 | 41,7 | 822 | 830 | 3,6 | 0,035 | 1,001 |
| | brez zareze | 2,6 | T = -40°C | Ø 8 | 8,0 / 5,75 | 50,27 / 25,97 | 40 | 47,25 | 36,27 | 37,50 | 722 | 746 | 5,0 | 0,0488 | 0,660 |
| | z zarezo | 2,7 | T = -40°C | Ø 8 | 5,6 / 5,4 | 24,63 / 22,90 | 40 | — | 27,6 | 33,8 | — | 1372 | 1,4 | 0,014 | — |
| | z zarezo | 2,8 | T = -40°C | Ø 8 | 5,56 / 5,0 | 24,28 / 19,63 | 40 | 43,7 | 30,1 | 30,1 | — | 1240 | 0,54 | 0,0054 | — |
| | brez zareze | 2,9 | T = -100°C | Ø 8 | 8,0 / 4,7 | 50,24 / 17,34 | 40 | 43,65 | 36,0 | 37,6 | 717 | 748 | 6,4 | 0,062 | 1,063 |
| | brez zareze | 2,10 | T = -100°C | Ø 8 | 8,0 / 4,7 | 50,24 / 17,34 | 40 | 49,1 | 38,48 | 39,80 | 766 | 792 | 5,83 | 0,0567 | 1,063 |
| | z zarezo | 2,11 | T = -100°C | Ø 8 | 5,6 / — | 24,62 / — | 40 | — | 30,6 | 30,6 | — | 1243 | 2,8 | 0,028 | — |
| Epruvete odvzete prečno na smer valjanja ≠ | z zarezo | 2,12 | T = -100°C | Ø 8 | 5,7 / 5,1 | 25,52 / 20,43 | 40 | 40,6 | 32,2 | 32,2 | — | 1262 | 0,75 | 0,00747 | — |
| | brez zareze | 2,13 | T = -150°C | Ø 8 | 8,0 / 5,10 | 50,27 / 20,43 | 40 | 41,2 | 37,89 | 39,0 | 754 | 776 | 1,46 | 0,0145 | 0,900 |
| | brez zareze | 2,14 | T = -150°C | Ø 8 | 8,0 / 5,20 | 50,27 / 21,24 | 40 | 44,65 | 43,2 | 43,2 | 859 | 859 | 3,3 | 0,0328 | 0,861 |
| | z zarezo | 2,15 | T = -150°C | Ø 8 | 5,69 / 5,30 | 25,4 / 22,05 | 40 | — | 35,3 | 35,3 | — | 1390 | 0,82 | | — |
| | z zarezo | 2,16 | T = -150°C | Ø 8 | 5,6 / 5,20 | 24,63 / 21,24 | 40 | 40,4 | 36,5 | 36,5 | — | 1482 | 0,17 | 0,00167 | — |
| | brez zareze | 2,17 | T = -196°C | Ø 8 | 8,0 / 5,7 | 50,27 / 25,52 | 40 | pretrg v m. tč. | 45,6 | 45,6 | 907 | 907 | 0,58 | 6,00582 | 0,677 |
| | brez zareze | 2,18 | T = -196°C | Ø 8 | 8,0 / 6,35 | 50,27 / 31,67 | 40 | 41,8 | 55,6 | 55,6 | 1106 | 1106 | 0,79 | 0,00789 | 0,462 |
| | z zarezo | 2,19 | T = -196°C | Ø 8 | 5,6 / 5,6 | 24,63 / 24,63 | 40 | 40,1 | 32,8 | 32,8 | — | 1332 | 0,21 | 0,00208 | — |
| | z zarezo | 2,20 | T = -196°C | Ø 8 | 5,7 / 5,6 | 25,52 / 24,63 | 40 | 40,1 | 39,1 | 39,1 | — | 1532 | 0,29 | 0,00291 | — |
| | z zarezo | 2,0 | T = -196°C | Ø 8 | 5,8 / 5,7 | 26,42 / | 40 | 40 | 33,2 | 33,2 | 1257 | 1257 | 0,21 | 0,00208 | — |
| | z zarezo | 2,22 | T = -196°C | Ø 8 | 5,8 / 5,5 | 26,42 / 23,76 | 40 | 40,1 | 36,5 | 36,5 | 1381 | 1381 | 0,33 | 0,00291 | — |

formabilnost jekla. Zarezno trdnostno razmerje se tudi ne spreminja kaj dosti s temperaturo preizkušanja, kar zlasti velja za dobavno stanje pločevine NIOMOL 490. Šele pri staranem stanju opažamo poslabšanje pri temperaturah preizkušanja, ki so nižje od −140°C. Zdi se tudi, da korenski radius zareze ni tako zelo odločilen, vsaj v tem primeru, saj je zarezno trdnostno razmerje staranega jekla, merjeno pri −196°C, skoraj enako, tako pri korenskem radiusu 0,25 mm (NSR = 1,423), kot tudi pri korenskem radiusu 0,025 mm (NSR = 1,311).

Temperatura prehoda v krhko stanje za statični režim obratovanja, merjena z enakomernim raztezkom v odvisnosti od temperature preizkušanja, je zanesljivo nižja od −140°C, čeprav je tedaj absolutna vrednost enakomernega raztezka že razmeroma nizka (velja za starano stanje, ne pa za material v dobavnem stanju), enako velja tudi za lomno duktilnost. Tudi razmerje med mejo elastičnosti in trdnostjo jekla je tedaj že zelo blizu 1, kar pomeni, da se tedaj jeklo obnaša že skoraj povsem elastično. Rekapitulacija mehanskih lastnosti NIOMOLA 490 ter izračunano zarezno trdnostno razmerje NSR sta razvidna iz **tabele X** ter grafično iz diagrama na **sliki 4**.

**Tabela 10:** Mehanske lastnosti Niomola 490 izmerjene pri statičnem preizkušanju

| Temper. preizk. °C | Rp N/mm² | Rm N/mm² | Razmerje Rp/Rm N/mm² | Največji enakomerni raztezek % | Trdnost zarezanega preizkuša. N/mm² | Zarezno trdnostno razmerje NSR |
|---|---|---|---|---|---|---|
| Niomol 490 — dobavno stanje | | | | | | |
| + 20 | 482 | 602 | 0.80 | 14.9 | 867 | 1.440 |
| − 40 | 553 | 694 | 0.79 | 15.5 | 1073 | 1.546 |
| −100 | 548 | 686 | 0.80 | 17.5 | 1116 | 1.626 |
| −150 | 657 | 791 | 0.83 | 15.6 | 1182 | 1.494 |
| −196 | 683 | 783 | 0.87 | 8.9 | 1393 | 1.779 |
| Niomol 490 — 10 % deformiran in staran 30'/250°C | | | | | | |
| + 20 | 655 | 672 | 0.97 | 3.6 | 1010 | 1.503 |
| − 40 | 772 | 788 | 0.98 | 4.3 | 1306 | 1.657 |
| −100 | 741 | 770 | 0.96 | 6.1 | 1252 | 1.626 |
| −150 | 806 | 817 | 0.98 | 2.4 | 1436 | 1.757 |
| −196 | 1006 | 1006 | 1.00 | 0.7 | 1432<br>1319+ | 1.432<br>1.311+ |

**Opomba:** Vrednosti označene s + se nanašajo na korenski radius zareze 0,025 mm, medtem, ko so vse ostale vrednosti veljavne za zarezo s korenskim radiusom 0,25 mm (Charpy-V zareza)

kjer pomeni:

$NSR = \frac{R_{m,net}}{R_{m,u}}$ zarezna krhkost oziroma občutljivost

Ker z zniževanjem temperature zarezno trdnostno razmerje NSR, kot tudi enakomerni raztezek $e_{gt}$ nekaj časa še rasteta in ker bi ta učinek lahko pripisali morebitno prisotnemu zaostalemu avstenitu v mikrostrukturi (posledica toplotne obdelave jekla) oziroma iz njega nastalem martenzitu pri nizkih temperaturah preizkušanja, smo iz NIOMOLA 490 izdelali 4 natezne preizkušance, od katerih sta bila dva preizkušena pri sobni temperaturi, dva pa sta bila podhlajena 1/2 ure pri T = −196°C (tekoči dušik), ogreta do sobne temperature, nato pa je bil opravljen natezni preizkus. Pri nateznem preizkusu nismo opazili nobenih razlik med obema vrstama preizkušancev, kar pomeni, da je imelo jeklo stabilno mikro-

*The obtained results prove the NIOMOL 490 steel does not show a substantial impairment in properties down to the temperature of −100°C. This steel properties in the as delivered condition improve considerably after strain aging, but the comparison of these properties in relation to testing temperature is practically the same.*

*It is evident, that the uniform elongation is a better indicator of ductility of the steel, as the notch strenght ratio. This is surprising, considering that the uniform elongation measures only the largest achievable homogenous deformation whilst the notch strength ratio measures also the propensity of strain hardening of the steel at the notch. Similarly the ductility at fracture is a good measure for the deformability of the steel. The test temperature does not change the notch strength ratio very much, which is particularly true for the as delivered condition. After strain aging an impairment is observed at test temperatures lower than −140°C. It seems that the radius at notch root is not decisive — at least in this case — as the notch strength ratio of the aged steel, measured at −196°C, is almost the same at the root radius of 0,25 mm (NSR = 1,423), as at the root radius of 0,025 mm (NSR = 1,311).*

*The transition temperature of the brittle state for the static loading, measured with the uniform elongation is certainly lower than −140°C although at this point the absolute value of the uniform elongation is already relatively low (this is valid for the aged, but not for the as delivered steel). The same can be said for the ductility at fracture. The ratio between the yield strength and tensile strenght is then already near 1, which means that in this case the steel behaves almost completely elastically. The recapitulation of the mechanical properties of NIOMOL 490 and the calculated notch strength ratio on NSR are shown in **table X**, and in diagram in **Figure 4**.*

***Table 10:** Mechanical properties of Niomol 490 evaluated by static load testing*

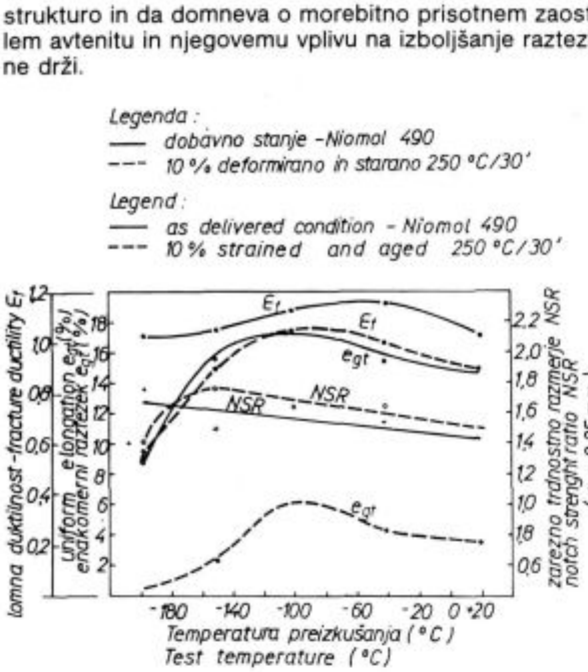
| Test temper. °C | Rp N/mm² | Rm N/mm² | Ratio-Rp/Rm N/mm² | Largest uniform elongation % | Tensile strength of notched specimen N/mm² | Notched strength ratio NSR |
|---|---|---|---|---|---|---|
| Niomol 490 — as delivered | | | | | | |
| + 20 | 482 | 602 | 0.80 | 14.9 | 867 | 1.440 |
| − 40 | 553 | 694 | 0.79 | 15.5 | 1073 | 1.546 |
| −100 | 548 | 686 | 0.80 | 17.5 | 1116 | 1.626 |
| −150 | 657 | 791 | 0.83 | 15.6 | 1182 | 1.494 |
| −196 | 683 | 783 | 0.87 | 8.9 | 1393 | 1.779 |
| Niomol 490 — 10 % strained and aged 30'/250°C | | | | | | |
| + 20 | 655 | 672 | 0.97 | 3.6 | 1010 | 1.503 |
| − 40 | 772 | 788 | 0.98 | 4.3 | 1306 | 1.657 |
| −100 | 741 | 770 | 0.96 | 6.1 | 1252 | 1.626 |
| −150 | 806 | 817 | 0.98 | 2.4 | 1436 | 1.757 |
| −196 | 1006 | 1006 | 1.00 | 0.7 | 1432<br>1319+ | 1.432<br>1.311+ |

***Remark:** Values marked with + refer to the root radius of the 0,025 mm notch, all other values are valid for a notch with a root radius of 0,25 mm (Charpy-V notch)*

*with the following equation:*

*Notch brittlenes sensitivity* $NSR = \frac{R_{m,net}}{R_{m,u}}$

strukturo in da domneva o morebitno prisotnem zaostalem avtenitu in njegovemu vplivu na izboljšanje raztezka ne drži.




**Slika 4.**
Enakomerni raztezek $e_{gt}$, lomna duktilnost $\varepsilon_f$ in zarezno trdnostno razmerje NSR v odvisnosti od temperature preizkušanja za NIOMOL 490 v dobavnem stanju in 10 % deformiranem ter staranem stanju (30 minut pri 250° C)

***Figure 4.***
*Uniform elongation $e_{gt}$, fracture ductility $\varepsilon_f$,* and the notch strength ratio NSR as a function of test temperature for NIOMOL 490 in the as delivered condition and for 10 % deformed and aged condition (30 minutes at 250° C)

## 3.2 Vpliv deformacije v hladnem ter staranja na žilavost jekla NIOMOL 490 in NIOMOL 490 K, Drop Weight Test

Da bi neposredno dokazali, da je jeklo NIOMOL 490 nagnjeno k staranju, smo opravili razbremenilni natezni preizkus, rezultati so razvidni s **slike 4**, merjenje Charpy-V žilavosti v odvisnosti od temperature pa je razvidno s **slike 5**.

Poleg opisanih raziskav smo določili še temperaturo neduktilnega loma NDT po standardu ASTM E 208. Rezultati vseh meritev so zbrani v **tabeli XI**.

Iz rezultatov je razvidno, da že manjša deformacija v hladnem povzroči zamik prehodne temperature k višjim vrednostim ($\Delta T = 55°$ C), da pa je zaradi procesov staranja ta zamik še intenzivnejši ($\Delta T = 83°$ C).

Staranje izhodnega materiala, ki ni bil predhodno deformiran v hladnem, ne poslabša bistveno prehodne temperature ($\Delta T = 15°$), čeprav so bili časi staranja dovolj dolgi (20 ur pri T = 250° C).

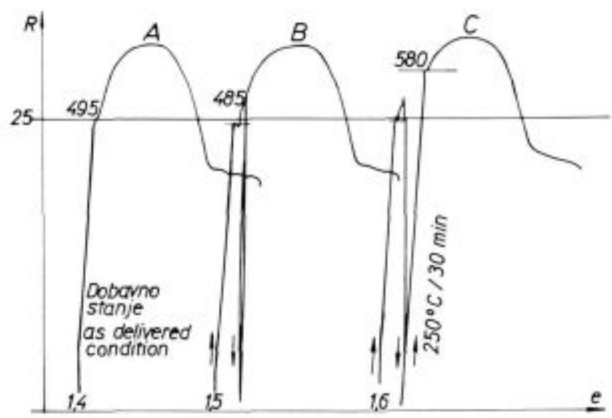
Omeniti moramo še vpliv ostrine zareze na Charpyevih preizkušancih. Evidentno je, da je preizkušanje z V zarezo ostrejše, presenetljiva pa je velika razlika med obema prehodnima temperaturama, kar $\Delta T = 40°$ C, kar je za prakso zelo pomembno spoznanje. Sledi namreč, da je neobhodno meriti žilavost tudi z metodami mehanike loma, torej z atomarno ostro zarezo, tako da zajamemo najmanj ugodne pogoje s stališča eksploatacije pri nizkih temperaturah.

Ker, kot že rečeno, za preiskano jeklo NIOMOL 490 ni bilo mogoče določiti korelacije med NDT-temperaturo ter temperaturo prehoda v krhko stanje tudi za starano jeklo, smo ta del preizkusov ponovili, in sicer z jeklom

*With decreasing temperature the notch strenght ratio NSR and the uniform elongation $e_{gt}$ increase for a short period of time which could be related to the presence of residual austenite, and to the martensite respectively, formed at lower test temperatures — four tensile test specimens were prepared from NIOMOL 490 to prove it. Two of them were tested at room temperature, the other two were cooled down to T = − 196° C in liquid nitrogen hold for 1/2 an hour, and tested at room temperature. The tensile test showed no differences between the two specimens. That means that the steel had a stable microstructure and that the supposition of presence of residual austenite is not founded.*

## *3.2 Strain aging of NIOMOL 490 and NIOMOL 490 K, checked by Drop Weight Test*

*To prove directly that the steel NIOMOL is affected by strain aging the unloading tensile test shown in **Figure 4** was performed while the effect of temperature on the Charpy-V impact toughness is shown in **Figure 5**.*




**Slika 5.**
Natezni preizkus NIOMOLA 490 (t = 12 mm) za ugotavljanje podvrženosti k staranju

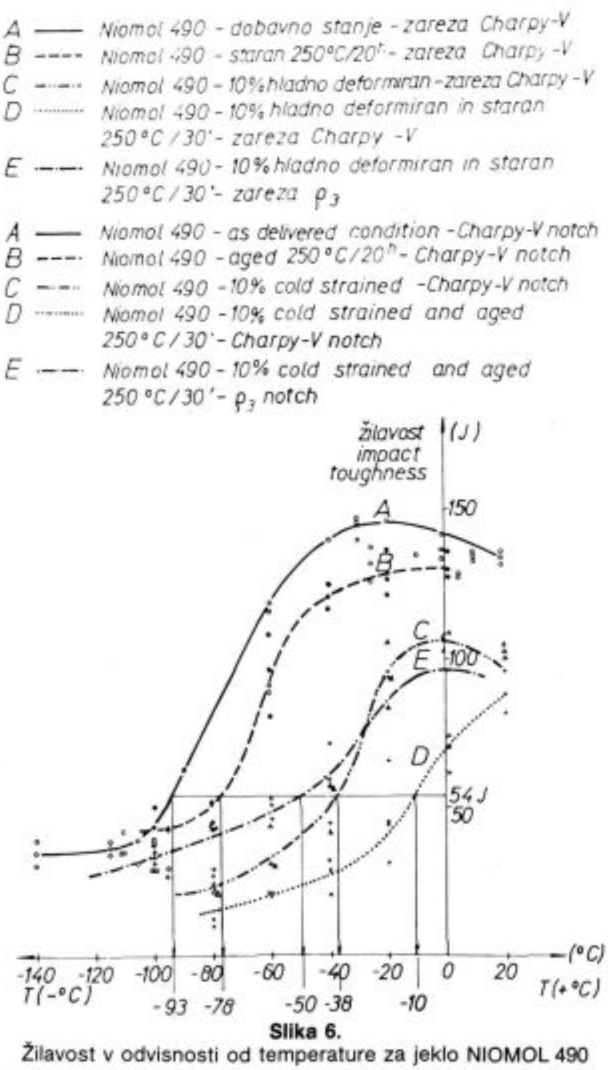
***Figure 5.***
*Tensile test of NIOMOL 490 (t = 12 mm) to ascertain proneness to aging*

***Table 11:*** *Results of measurements performed for the Niomol 490 steel*

| *Designation of the curve* | *Steel aging* | *Type of notch on specimen* | *Measured transition temperat T54 (°C)* | *Drop weight test results (°C)* |
|---|---|---|---|---|
| A | initial condition/ normalized with a ferrit-bainit structure | Charpy V | − 93 | − 85 |
| B | initial condition + aging 250° C/20 hours | Charpy V | − 78 | — |
| C | initial condition + cold straining | Charpy V | − 38 | — |
| D | initial condition + 10 % cold straining + aging 250° C/30 min | Charpy V | − 10 | — |
| E | initial condition + 10 % straining aging 250° C/30 min | Charpy U ($\rho_\varepsilon$) | − 50 | — |

**Tabela 11:** Rezultati meritev za jeklo Niomol 490

| Oznaka krivulje | Staranje jekla | Tip zareze na vzorcu | Izmerjena tempera-tura prehoda T54 (°C) | Vrednosti drop weight testa (°C) |
|---|---|---|---|---|
| A | izhodno stanje (normalizirano s feritno-bainitno mikrostrukturo) | Charpy V | −93 | −85 |
| B | izhodno stanje + staranje 250°C/20 ur | Charpy V | −78 | — |
| C | izhodno stanje + hladna deformacija | Charpy V | −38 | — |
| D | izhodno stanje + 10 % deformacija v hladnem + staranje 250°C/30 minut | Charpy V | −10 | — |
| E | izhodno stanje + 10 % deformacija v hladnem + staranje 250°C/30 minut | Charpy U ($\rho_E$) | −50 | — |




**Slika 6.**
Žilavost v odvisnosti od temperature za jeklo NIOMOL 490
***Figure 6.***
*Impact toughness as a function of temperature for the NIOMOL 490*

*The nill ductility temperature NDT as defined in ASME E 208 was determined. The results of all measurements are shown in **table XI**.*

*It is evident, that already a small cold straining causes the transition temperature to shift to higher values ($\Delta T = 55°C$), and that this shift is still greater after strain aging ($\Delta T = 83°C$). The aging of the initial material which previously was not cold strained does not essentially affect the transition temperature ($\Delta T = 15°C$) not even after a relatively long aging time up to 20 hours at $T = 250°C$.*

*The influence of the notch sharpness on Charpy specimens should be mentioned, too. It is evident, that the test with the V notch is more rigorous. The difference between the two transition temperatures, as high as $\Delta T = 40°C$ is surprising and very important from the user standpoint. It suggests namely that the toughness must be measured by the methods of fracture mechanics i. e. by an atomic sharp notch considering the least favourable conditions of use at low temperature.*

*As already mentioned, for the investigated NIOMOL 490, it was not possible to determine the correlations between NDT and the transition temperatures not even for the aged steel, therefore these tests were partly repeated with NIOMOL 490 K, which is a modified version of NIOMOL 490, with a transition temperature shifted to a lower degree. The results are shown in Figure 6 and in **table XII**.*

***Table 12:*** *Correlation between transition temperatures into brittle state by the 54 J, 68 J criteria, resp. NDT temperature from DWT for Niomol 490 k*

| Material | Transition temp. to brittle state | | NDT temperature from NWT |
|---|---|---|---|
| | Criterion 54 J | Criterion 68 J | |
| | | (°C) | |
| A | −122 | −115 | −120 |
| B | −112 | −110 | −105 |
| C | − 80 | − 77 | − 78 |

*A Niomol 490 K — initial state, that is as delivered*
*B Niomol 490 K — aged state (250°C/30 minutes)*
*C Niomol 490 K — 10 % cold strained and aged (250°C/30 minut)*

*Temperature of the transition to the brittle state on the basis of the average value for impact toughness 54 J (lit.[1]) for NIOMOL 490, and on the basis of the criteria 68 J (lit.[2]) for steels of similar type were determined from diagram in **Figure 6**. The curves in the transition region are very steep, thus the reading is very approximative. The obtained values are shown together with the NDT temperatures of drop weight tests in **table XII**.*

*The conformity of the Charpy's temperature of transition into brittle state to the NDT of drop weight tests is surprising. It is important for the application of NIOMOL 490 K, but at the same time the general validity of the reference value of the CAT curve by Pellini is questioned. Namely, it is obvious that the relation between NDT and Charpy transition temperatures (into the brittle state) is complicated and varies from type to type of steel.*

### *3.3 Microfractografic examinations*

***Figure 7*** *shows the morphology of the fracture surface of a NIOMOL 490 Charpy-V specimen in the initial*

NIOMOL 490 K, torej z modificirano verzijo jekla NIOMOL, ki pa ima temperaturo prehoda pomaknjeno k nižjim vrednostim. Rezultati so razvidni s **slike 6** in **tabele XII**.

**Tabela 12:** Korelacija med temperaturo prehoda v krhko stanje po kriteriju 54 J, 68 J ter NDT temperaturo DWT testa

| Material | Temperatura prehoda v krhko stanje | | NDT temperatura iz DWT testa |
|---|---|---|---|
| | Kriterij 54 J | Kriterij 68 J | |
| | (°C) | | |
| A | −122 | −115 | −120 |
| B | −112 | −110 | −105 |
| C | − 80 | − 77 | − 78 |

A Niomol 490 K — osnovno, to je dobavno stanje
B Niomol 490 K — starano stanje (250 °C/30 minut)
C Niomol 490 K — 10 % deformirano v hladnem in starano (250° C/30 minut)

Iz diagrama na **sliki 6** je odčitana temperatura prehoda v krhko stanje na osnovi kriterija srednje vrednosti žilavosti 54 J (lit.[1]), kot pri NIOMOLU 490, in na osnovi kriterija 68 J (lit.[2]) za jekla podobne vrste. Krivulje so namreč v območju temperature prehoda tako strme, da je izbira kriterija lahko poljubna. Dobljene vrednosti so skupaj z NDT temperaturami Drop Weight Testa zbrane v **tabeli XII**.

Ujemanje med Charpyjevimi temperaturami prehoda v krhko stanje ter NDT temperaturami je presenetljivo. S stališča uporabe jekla NIOMOL 490 K je to sicer zelo pomembno, vendar pa se ob tem postavlja vprašanje splošne veljavnosti definiranih referenčnih vrednosti na CAT krivulji po Pelliniju. Očitno je namreč, da je odvisnost med NDT ter Charpyjevimi temperaturami prehoda v krhko stanje bolj komplicirana in tudi povsem različna za različne vrste jekel.

### 3.3 Mikrofraktografske preiskave

**Na sliki 7** je prikazana morfologija preloma površine Charpy-V preizkušanca jekla NIOMOL 490 je v izhodnem nestaranem stanju pri temperaturi −80° C.

Poleg obsežnih področij cepilnega tipa loma opazimo tudi področja jamičaste duktilne ločitve, ki jeklu še tudi pri tako nizki temperaturi preizkušanja dajejo znatno žilavost (80 J).

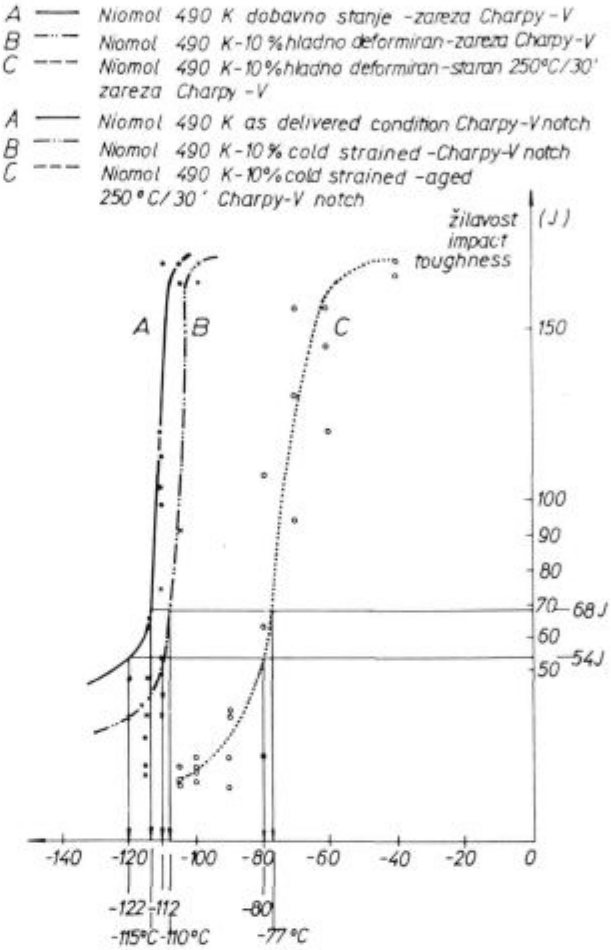
Mikromorfologija prelomne površine Charpy-V preizkušanca, izdelanega iz staranega jekla (10 % deformacije v hladnem +250° C/30 minut) pri T = −80° C je prikazana na **sliki 8**.

Prelom je povsem cepilne narave in temu je ustrezna tudi žilavost, vsega 20 J. V nasprotju s preizkušancem z V zarezo pa na preizkušancu z U zarezo, preizkušanim pod enakimi pogoji, lahko še vedno najdemo tudi področja duktilne ločitve, kot je to videti s **slike 9**.

Na Charpy-V preizkušancih iz staranega jekla se prvi duktilni grebeni pričnejo pojavljati na frakturnih površinah šele pri temperaturi preizkušanja −40° C ali višji, kot je to razvidno s **slike 10**.

Podobna kot **slika 10** je tudi **slika 11**, ki pa prikazuje prelomno površino Charpy-V preizkušanca iz jekla, ki je bilo le deformirano v hladnem za 10 %, ne pa tudi starano.

Prvi duktilni grebeni se tu pojavljajo pri temperaturi preizkušanja −60° C ali večji. **(slika 12)**



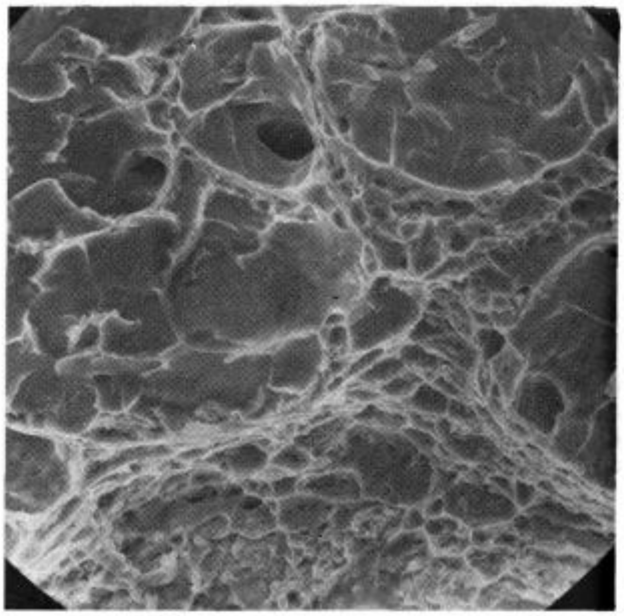

**Slika 7.**
Žilavost Charpy-V v odvisnosti od temperature za jeklo NIOMOL 490 K

***Figure 7.***
*Charpy-V impact toughness as a function of temperature for the NIOMOL 490 K*

*(not aged) condition at the temperature − 80° C. Next to the extensive areas of cleavage fracture areas of dimpled and ductile decohesion make steel considerably tough (80 J) at very low test temperatures.*

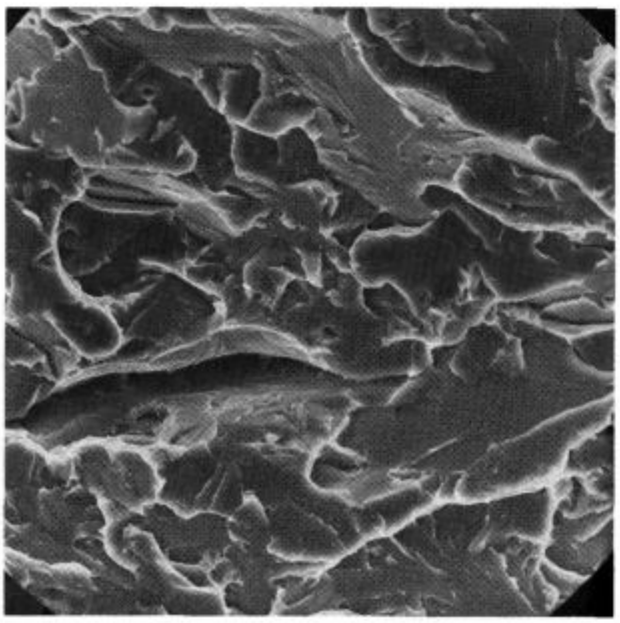
***Figure 8*** *shows the fracture surface micromorphology of a Charpy-V specimen of a strain aged steel at T= 80°. The fracture surface is of a cleavage type and consequently, the toughness amounts to only 20 J. Contrary to the specimen with the V notch, in the U notch specimen under the same conditions, areas of ductile propagation still can be found, as shown in* ***Figure 9***.

*On the fracture surface of the Charpy V specimens made from aged steel the first ductile ridges appear at the test temperature of − 40° C or higher, as shown in* ***Figure 10***. *Like* ***Figure 10, Figure 11*** *shows a fracture surface of a Charpy-V specimen of a steel, which was cold strained for 10 % but not aged. The first ductile ridges appear in this case at a test temperature of − 60° C or higher. Finally it can be established that the nature of the fracture of the examined Charpy-V specimens in the region of transition temperature is of a mixed type. Besides, cleavage facets on the smaller or larger areas of ductile fracture can be distinguished. These areas essentially contribute to the toughness because the toughness of the completely cleavage fracture is nearly nil.*



**Slika 8.**
Mikromorfologija preloma površine Charpy-V preizkušanca jekla NIOMOL 490 v izhodnem stanju pri T = −80° C

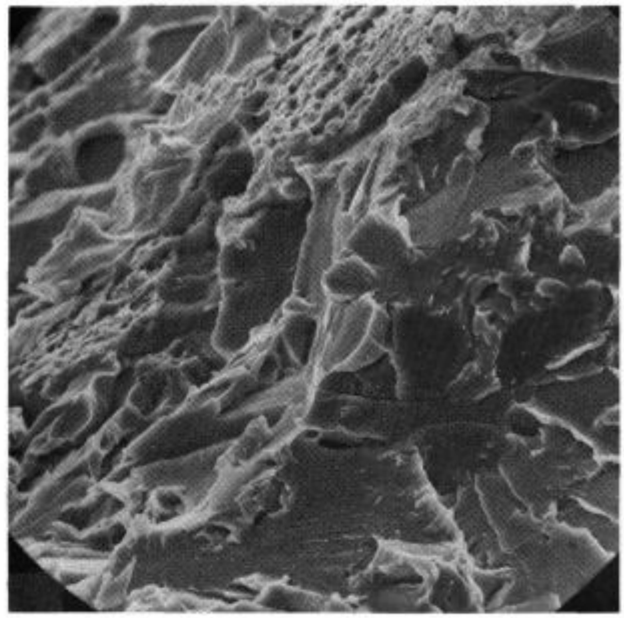
***Figure 8.***
*Micromorphology of the fracture surface on Charpy-V specimen of the NIOMOL 490 in the initial condition at T = −80° C*



**Slika 9.**
Mikromorfologija preloma površine Charpy-V preizkušanca jekla NIOMOL 490 za starano jeklo (10 % def. v hladnem + 250° C/30 min.) pri T = −80° C

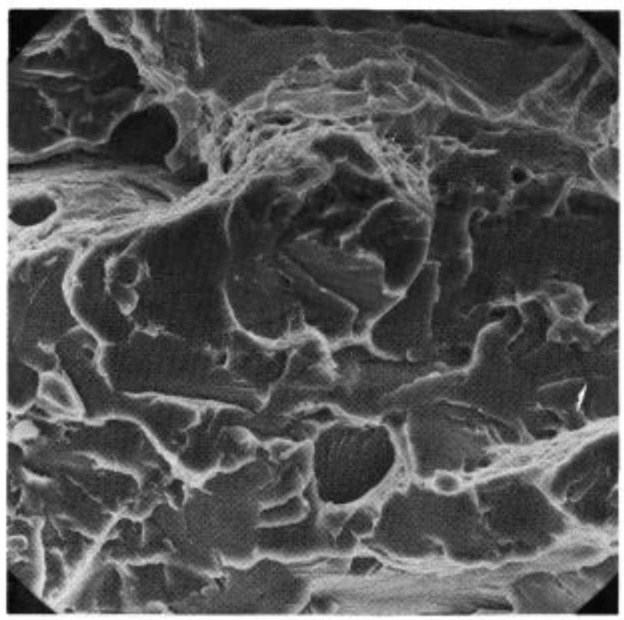
***Figure 9.***
*Fracture surface micromorphology of Charpy-V specimen of the aged NIOMOL 490 (10 % cold deformation and + 250° C/ 30 min.) at T = −80° C*

Zaključimo lahko z ugotovitvijo, da je narava preloma preiskanih CHARPY-V preizkušancev v območju prehodnih temperatur mešane oblike. Poleg cepilnih ploskev je na posnetkih moč razločiti večja ali manjša področja jamičastega duktilnega tipa loma. Žilavost jekla dajejo, oziroma k njej prispevajo, le ta duktilna področja.



**Slika 10.**
Mikromorfologija preloma površine Charpy-U preizkušanca jekla NIOMOL 490 za starano jeklo (10 % def. v hladnem + 250° C/30 min.) pri T = −80° C

***Figure 10.***
*Fracture surface micromorphology of Charpy-U specimen of the aged NIOMOL 490 (10 % cold deformation and aged 250° C/ 30 minutes) at T = −80° C*

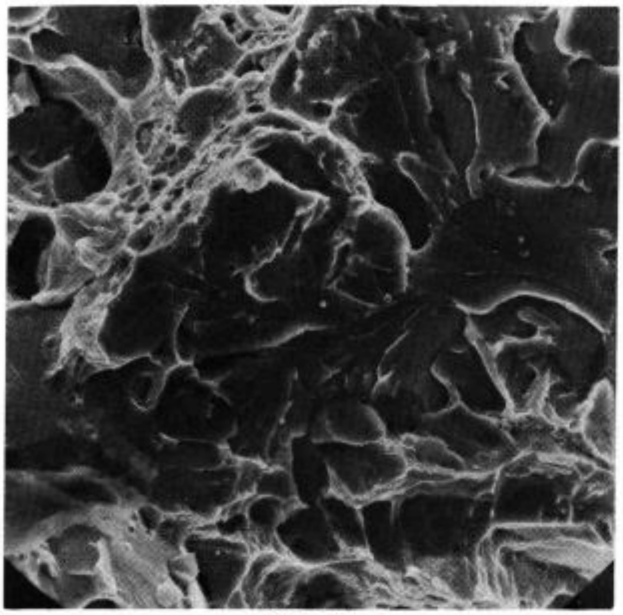


**Slika 11.**
Mikromorfologija preloma površine Charpy-V preizkušanca jekla NIOMOL 490 za starano jeklo (10 % def. v hladnem + starano 250° C/30') pri T = −40° C

***Figure 11.***
*Fracture surface micromorphology of Charpy-V specimen of the NIOMOL 490 for the aged steel (10 % cold deformation and 250° C/30 minutes) at −40° C*

### *3.4 Application of fracture mechanics to the analysis of results*

*The analysis was performed on NIOMOL 490 in the as delivered condition because this steel's transition to*



**Slika 12.**
Mikromorfologija preloma površine Charpy-V preizkušanca iz jekla NIOMOL 490 v nestaranem stanju (le 10 % def. v hladnem) pri T= −60°C

***Figure 12.***
*Fracture surface micromorphology of Charpy-V specimen of the not aged NIOMOL 490 (10 % cold deformation only) at T= −60°C*

medtem ko je žilavost s povsem cepilno obliko loma praktično nična.

### 3.4 Uporaba lomne mehanike pri analizi rezultatov

Za analizo je bilo izbrano jeklo NIOMOL 490 v dobavnem stanju. Pri tem jeklu je namreč prehod v krhko stanje bolj položen, zato je računanje soodvisnosti med temperaturami prehoda in NDT temperaturami sploh smiselno. Pri temperaturi −20°C (253 K) je bila žilavost Charpy-V tega jekla enaka 145 J. Lomno žilavost $K_{IC}$ izračunamo najprej s korelacijo Barsom-Rolfe za **območje prehodnih temperatur**, kot so navajata Faucher in Dogan (lit.[1]):

$$K_{IC}^2 = 0{,}22 \cdot E \cdot CVN^{1.5}$$

Dobimo:

$$K_{IC}^2 = 0{,}22 \cdot 2{,}05 \cdot 10^2 \cdot 145^{1.5}$$

$$K_{IC} = 280 \text{ MPa } (m)^{1/2}$$

Pri tem smo za modul elastičnosti E vstavili vrednost $2{,}05 \cdot 10^2$ GN $m^{-2}$, Charpyjeva-V žilavost pa je izražena v joulih.

Nekoliko nižjo vrednost dobimo, če izračunamo lomno žilavost $K_{IC}$ s pomočjo Hahn-Rosenfieldove korelacije. Dobimo namreč:

$$K_{IC} = (0{,}05 \cdot \varepsilon_f \cdot n^2 \cdot E \cdot R_p/3)^{1/2}$$

$$K_{IC} = (0{,}05 \cdot 1{,}20 \cdot 0{,}149^2 \cdot 2{,}05 \cdot 10^5 \cdot 472/3)^{1/2}$$

$$K_{IC} = 207 \text{ MPa } m^{1/2}$$

Če izračunamo še $K_{IC}$, kot sledi na osnovi merjenja J integrala (lit.[4]), dobimo za $K_{IC}$ rezultat ($K^2 = J \cdot E$):

$$K_{IC}^2 = 655 \cdot 2{,}05 \cdot 10^8 \text{ kN}^2 \text{ m}^{-3}$$

$$K_{IC} = 336 \text{ MPa } m^{1/2}$$

Na osnovi izračunavanj lahko sklepamo, da bi morebiti lahko $K_{IC}$ vrednosti pri nizkih temperaturah določili

*brittle state is slower, therefore calculation of the correlation between transition temperature and NDT temperatures is meaningful at all. At the temperature of −20°C (253 K) the Charpy V toughness of this steel was 145 J. The fracture toughness $K_{IC}$ is obtained first from the Barsom-Rolfe correlation for the transition temperature region, as stated by Faucher and Dogan (Ref.[1]):*

$$K_{IC}^2 = 0{,}22 \cdot E \cdot CVN^{1.5}$$

*which shows:*

$$K_{IC}^2 = 0{,}22 \cdot 2{,}05 \cdot 10^2 \cdot 145^{1.5}$$

$$K_{IC} = 280 \text{ MPa } (m)^{1/2}$$

*For the elastic modulus the value of $2{,}05 \cdot 10^2$ GN $m^{-2}$ was chosen and the Charpy V notch toughness expressed in Joules. Lower values are obtained if the fracture toughness $K_{IC}$ is calculated with the Hahn-Rosenfield correlation:*

$$K_{IC} = (0{,}05 \cdot \varepsilon_f \cdot n^2 \cdot E \cdot R_p/3)^{1/2}$$

$$K_{IC} = (0{,}05 \cdot 1{,}20 \cdot 0{,}149^2 \cdot 2{,}05 \cdot 10^5 \cdot 472/3)^{1/2}$$

$$K_{IC} = 207 \text{ MPa } m^{1/2}$$

*If now $K_{IC}$ is calculated on the basis of J integral measurement (lit.[4]) for $K_{IC}$, is the following:*

$$K_{IC}^2 = 655 \cdot 2{,}05 \cdot 10^8 \text{ kN}^2 \text{ m}^{-3}$$

$$K_{IC} = 336 \text{ MPa } m^{1/2}$$

*These calculations show that $K_{IC}$ could be determined at low temperatures simply with the correlation given by Rolfe-Novak and thus the relationship between $K_{IC}$ and NDT obtained. The values for NIOMOL 490 and a temperature $T_{NDT} = -85°C$ are shown in **table XIII**. The $K_{IC}$ values were calculated on the basis of the correlation given by Barsom-Rolfe as a function of the reference transition temperature of nil ductility $T_{NDT,ref}$ which is defined as the difference between the test temperature and the nil ductility temperature NDT.*

***Table 13:*** *Relationship between fracture toughness $K_{IC}$ and $T_{NDT,ref}$ for the Niomol 490 steel in the delivered state ($T_{NDT} = -85°C$)*

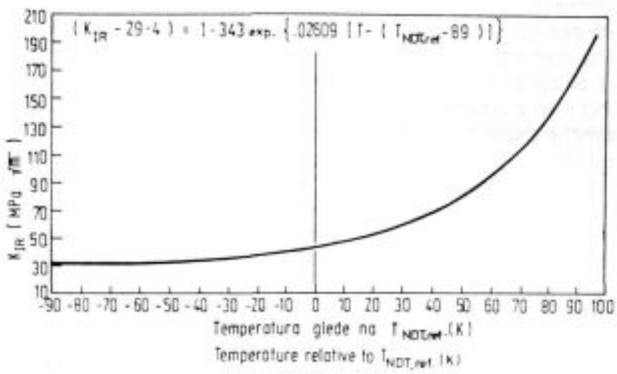
| *Test temperature (°C)* | *$T_{NDT,ref}$ (K)* | *Impact toughness Charpy-V (J)* | *Calculated fracture toughness $K_{IC}$ (Mpa$\sqrt{m}$)* |
|---|---|---|---|
| −20 | 65 | 145 | 280 |
| −60 | 25 | 120 | 243 |
| −75 | 10 | 100 | 212 |
| −85 | 0 | 68 | 159 |

*Let us compare the above mentioned results with the results (12, 13) from **Figure 13**, which shows the relationship between the reference values of fracture toughness (the lower band of these values) and the relative temperature as quoted above. The diagram is valid for steels, which are used in the USA for nuclear reactor pressure vessels. On the basis of this diagram two steels of similar type, which have different NDT temperatures can be compared resulting in a logical supposition is that at a given working temperature the resistance to fracture, defined by $K_{IC}$, differs. One of the two steels could be used at lower temperatures than the other, but with the same safety margin against fracture. As reference a value of $K_{IC}$ 210 MPa $\sqrt{m}$, is chosen. In the NIOMOL steel such a toughness is achieved at the working temperature, which is only 10°C higher than NDT so already in the hazardous area. Just the opposite hap-*

kar enostavno s korelacijo po Rolfe-Novak ter na ta način dobili soodvisnost med $K_{IC}$ in NDT. Za jeklo NIOMOL 490 v dobavnem stanju dobimo pri $T_{NDT} = -85°C$ vrednosti, podane v **tabeli XIII**.

**Tabela 13:** Soodvisnost med lomno žilavostjo $K_{IC}$ in $T_{NDT,ref}$ za jeklo Niomol 490 v dobavnem stanju ($T_{NDT} = -85°C$)

| Temperatura testiranja (°C) | $T_{NDT,ref}$ (K) | Žilavost Charpy-V (J) | Izračunana lomna žilavost - $K_{IC}$ (Mpa$\sqrt{m}$) |
|---|---|---|---|
| −20 | 65 | 145 | 280 |
| −60 | 25 | 120 | 243 |
| −75 | 10 | 100 | 212 |
| −85 | 0 | 68 | 159 |




**Slika 13.**
Spodnja meja pasu referenčnih $K_{IR}$ vrednosti lomne žilavosti glede na referenčno prehodno temperaturo ničelne žilavosti ($T - T_{NDT,ref}$)

***Figure 13.***
*Lower bound of $K_{IR}$ reference values as a function of the relative test temperature ($T - T_{NDT,ref}$)*

V tej tabeli so prikazane $K_{IC}$ vrednosti izračunane na osnovi korelacije Barsom-Rolfe v odvisnosti od referenčne prehodne temperature ničelne žilavosti $T_{NDT,ref}$. Torej je $T_{NDT,ref}$ določena kot razlika med temperaturo nične duktilnosti NDT in temperaturo testiranja.

Primerjamo sedaj zgornje rezultate z rezultati (lit.[5, 12]) s **slike 13**.

Iz diagrama na **sliki 13** izhaja soodvisnost med referenčnimi vrednostmi lomne žilavosti (spodnji pas teh vrednosti) in relativno temperaturo, kot je bila definirana zgoraj. Diagram je veljaven za jekla, ki se v ZDA uporabljajo za nuklearne reaktorske posode. Na osnovi tega diagrama lahko primerjamo dve jekli podobne vrste, ki imata različni NDT temperaturi. Logičen je namreč sklep, da je pri dani temperaturi eksploatacije odpornost teh dveh jekel proti lomu, določena s $K_{IC}$, različna. Eno od obeh jekel bo namreč uporabno do nižjih temperatur kot drugo, a z enako stopnjo zaščite pred lomom.

Kot referenčno vrednost za $K_{IC}$ izberemo npr. 210 MPa $\sqrt{m}$. Pri NIOMOLU je takšna žilavost dosežena pri temperaturi eksploatacije, ki je le še 10°C višja od NDT, torej smo že v nevarnem območju. Nasprotno pa je v primeru jekla s **slike 13**, kjer je takšna žilavost dosežena pri temperaturi eksploatacije, ki je kar za 100°C višja od NDT in smo zato od NDT varno oddaljeni.

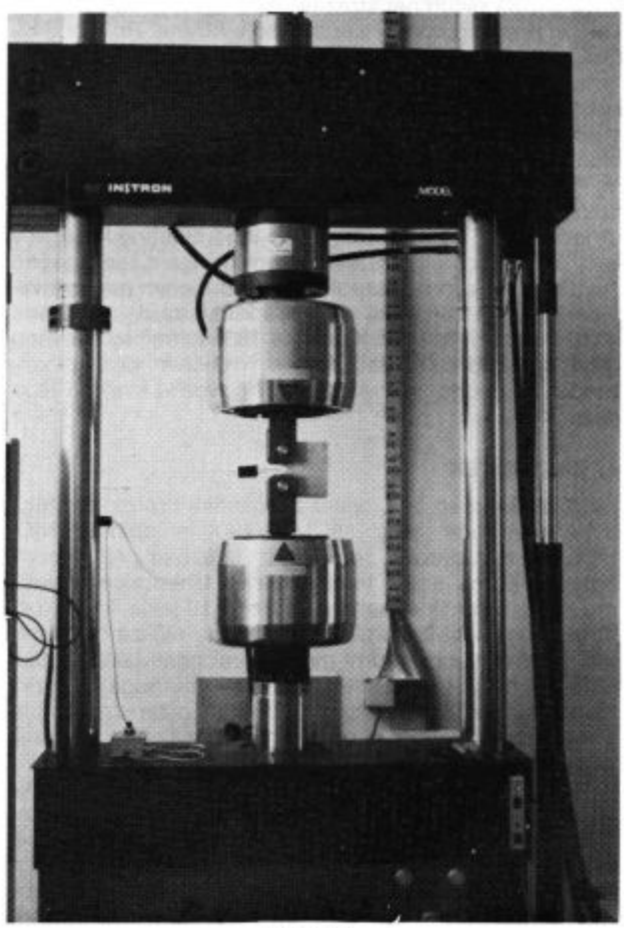
### 3.5 Določevanje lomne žilavosti jekla NIOMOL 490 z J integralom

J integral jekla NIOMOL 490 smo določili s CT preizkušanci po metodi ASTM E 813 (lit.[13]) in postopku, ki ga

*pens to the steel of **Figure 13** where such a toughness is reached at a working temperature of 100°C above the NDT in safe distance from NDT.*

### *3.5 Fracture toughness determination for NIOMOL 490 by J integral measurements*

*J integral of the NIOMOL 490 was measured with CT specimens according to ASTM 813 method (13) and a procedure recomended by J. Heerens and K. H. Schwalbe (14). The determination of J integral and $J_{IC}$ values was carried out by the method of one specimen with partial unloading. For detailed description of NIOMOL 490 testing see (Ref. 11). **Figure 14** shows a loaded CT specimen with an instaled clip gauge for load line displacement measurement. In **table XIV** the $J_{IC}$ values obtained by the two methods are listed. The results show that the classical determination of the J integral in accordance with the standard ASTM E 813 is unsuitable for such tough materials as NIOMOL 490 as the values obtained are unrealistically high for $J_{IC}$. The major part of the error is caused by blunting line which is unsuitable and does not consider the strain hardening of the metal. In the modified method proposed by J. Heerens, J. K. Schwalbe, the improvements refer to the R-line, which was aproximatec by an exponential function of the J form A. $(\Delta a)^B$ (A and B being constants, $\Delta a$ is*




**Slika 14.**
Obremenjen CT preizkušanec z montiranim merilcem hoda v liniji delovanja obremenitve

***Figure 14.***
*Loaded CT specimen with an installed clip gauge for load line displacement measurements*

**Tabela 14:** Vrednosti $J_{IC}$ določene po metodi ASTM E 813 in modificirani metodi J. Heerens, K. H. Schwalbe za jeklo Niomol 490

| Oznaka preizkušanca | $J_{IC}$ določen po Standardu ASTM E 813 | $J_{IC}$ določen po Modificirani metodi J. Heerens, K. H. Schwalbe |
|---|---|---|
| | kJ/m² | |
| 2.2 | — | 795 |
| 2.3 | 1135 | 720 |
| 2.5 | 960 | 655 |
| 2.6 | 1400 | 980 |
| 2.7 | 1520 | 870 |
| 2.9 | 1690 | 1330 |

priporočajo J. Heerens, K. H. Schwalbe (lit.[14]). Določanje J integrala in $J_{IC}$ vrednosti je potekalo po metodi z uporabo enega preizkušanca z delnim razbremenjevanjem. Potek preizkušanja NIOMOL 490 je podrobneje opisan v (lit.[11]). Na **sliki 14** je prikazan obremenjen CT preizkušanec z montiranim clip-gauge v liniji delovanja obremenitve. V **tabeli XIV** pa so zbrane vrednosti $J_{IC}$, določene po obeh metodah.

Iz rezultatov lahko povzamemo, da je klasično določanje J integrala skladno s standardom ASTM E 813, za tako žilave materiale, kot je NIOMOL 490, neustrezno, saj daje nerealno visoke vrednosti za $J_{IC}$. Največji del napake gre na račun neustrezne blunting linije (linije otopitve), ki premalo upošteva utrjevanje kovine. Pri modificirani metodi, ki jo predlagajo J. Heerens, J. K. Schwalbe . . . se izboljšave nanašajo tako na R linijo, ki smo jo aproksimirali s potenčno funkcijo oblike $J = A \cdot \Delta a^B$ (A, B sta konstanti, $\Delta a$ je napredovanje razpoke), kot tudi na blunting linijo, ki je določena z enačbo $\Delta a_B = 0{,}4 \cdot d_n \cdot J/\sigma_o$. Tako napetost tečenja $\sigma_o$, kot tudi faktor $d_n$ sta bila določena z upoštevanjem eksponenta deformacijskega utrjevanja n. Takšna realnejša enačba blunting linije, ki v večji meri upošteva utrjevanje kovine ter s tem močno plastifikacijo korena razpoke še pred njenim napredovanjem, daje v splošnem nižje vrednosti za $J_{IC}$. Dobljene vrednosti za $J_{IC}$ od 655 kJ/m² do 1330 kJ/m² so bistveno nižje od vrednosti, dobljenih s klasičnim postopkom, vendar še vedno zelo visoke, kar govori o kvaliteti tega jekla.

## 4. ZAKLJUČEK

Raziskane so bile lomne značilnosti drobnozrnatega mikrolegiranega jekla NIOMOL 490 in deloma NIOMOL 490 K. Ugotovili smo, da z nateznim preizkusom, opravljenim pri nizkih temperaturah, lahko sicer določimo konvencionalne mehanske lastnosti jekla, ki jih konstruktor potrebuje za dimenzioniranje, nič pa ne moremo soditi o temperaturni meji uporabnosti jekla. O tem nam nič ne pove niti merjenje enakomernega raztezka niti lomne duktilnosti in tudi ne merjenje zarezne občutljivosti. Vsa ta merjenja so namreč opravljena v statičnih pogojih.

Udarni preizkusi so zajeli tako študij vpliva staranja jekla na temperaturo prehoda v krhko stanje, določeno s Charpy-V merjenjem žilavosti, kot tudi določanje temperature nične duktilnosti jekla z Drop Weight Testom. Ugotovili smo, da se temperatura prehoda v krhko stanje, določena z merjenjem Charpy-V žilavosti, izredno dobro ujema s temperaturo ničelne duktilnosti jekla, kar daje pri tem jeklu merjenju Charpy-jeve žilavosti povsem nov pomen.

Mikrofraktografske preiskave so pokazale, da je prehod v krhko stanje povezan s spremembo morfologije preloma žilavostnih preizkušancev, katerih frakturna po-

*Table 14: Values of $J_{IC}$ determined in accordance with ASTM E 813 and the modified method by J. Heerens, K. H. Schwalbe for the Niomol 490 steel*

| Designation of the test specimen | $J_{IC}$ determined by Standard ASTM E 813 | $J_{IC}$ determined by Modified method by J. Heerens, K. H. Schwalbe |
|---|---|---|
| | kJ/m² | |
| 2.2 | — | 795 |
| 2.3 | 1135 | 720 |
| 2.5 | 960 | 655 |
| 2.6 | 1400 | 980 |
| 2.7 | 1520 | 870 |
| 2.9 | 1690 | 1330 |

*crack increasement), as well as to the blunting, which is defined with the equation $\Delta a_B = 0{,}4 \cdot d_n \cdot J/\beta o$. The flow stress $\beta o$ and also the factor $d_n$ were determined by taking into consideration the exponent of strain hardening n. Such a more realistic equation of the blunting line taking into consideration the strain hardening and thus the strong plastification of the cracks root before its propagation presents in general a lower value for $J_{IC}$. The obtained values for $J_{IC}$ from 655 kJ/m² to 1330 kJ/m² are essentialy lower from values obtained by a classical procedure, but still very high, which speaks for the quality of this steel.*

## 4. CONCLUSION

*Finegrained microalloyed steel NIOMOL 490 and partly NIOMOL 490K were investigated for fracture characteristics. It was established that by the tensile test performed at low temperatures the conventional mechanical properties required by designers can be determined, but nothing can be concluded on the temperature limits of the steel. Neither the uniform elongation nor the fracture ductility or notch sensibility give reliable information because all those measurements are performed under static conditions.*

*The impact tests included the research on the effects of strain aging on the transition temperature to brittle state determined by Charpy-V toughness measurements as well as the determination of nil ductility temperatures with the drop weight test.*

*It was established, that the transition temperature defined by measurements of Charpy-V notch toughness agrees well with the nil ductility temperature, which gives this steel a new meaning to Charpy-V notch impact toughness measurements.*

*Micro fractographic examinations showed that the transition in to brittle state is connected with the change in morphology of the fracture surface on impact toughess specimens which is a dimpled ductile in the upper shelf region. In the region of transition to brittle state the number of cleavage facets increases until at temperatures low enough the fracture becomes a completely brittle cleavage. The correlation between the fracture toughness of steel at a particular operating temperature and the reference nil ductility temperature, $T_{NDT,ref}$ is of vital importance for safe dimensioning, for NIOMOL 490 the correlation between fracture toughness (in the region of transition temperatures $K_{IC}$ calculated as proposed by Rolfe-Barson) and the reference nil ductility temperature, $T_{NDT,ref}$ was determined.*

*It became evident that just such a correlation is necessary for safe application of this steel at low temperatures. This makes possible a comparison with other* vršina je v območju »upper shelf« vrednosti povsem jamičasto duktilna, v območju prehoda v krhko stanje pa se povečuje delež cepilnih prelomnih ploskvic, vse dokler ni pri dovolj nizkih temperaturah prelom povsem cepilen.

Ker je za konstruktorja s stališča varnega dimenzioniranja izredno pomembna soodvisnost med lomno žilavostjo jekla pri določeni temperaturi eksploatacije in referenčno temperaturo ničelne duktilnosti jekla ($T_{NDT,ref}$), smo za preiskano jeklo NIOMOL 490 določili še odvisnost lomne žilavosti jekla (v območju prehodnih temperatur je bila $K_{IC}$ izračunana po Rolfe-Barsom) od referenčne prehodne temperature nične duktilnosti $T_{NDT,ref}$.

Izkazalo se je, da prav takšna soodvisnost pove vse o varni uporabi tega jekla pri nizkih temperaturah. Na tej osnovi je namreč možna primerjava s kakšnim drugim jeklom podobne vrste, ki pa ima drugačno NDT temperaturo; ugotovljena soodvisnost pa nam pove, katero od obeh bo imelo boljšo varnost proti krhkemu lomu.

*steels of similar type, but of a different NDT temperature. The determined correlation shows which of the two resists the brittle fracture the best.*

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