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## Žilavost osnovnega materiala in zvarov P91

### Toughness of P91 Base and Weld Metal

#### Povzetek

Jeklo P91 se izdatno uporablja v procesih pri povišanih temperaturah na področju fosilnih goriv in v jedrskih elektrarnah, in sicer za cevi in napeljave, saj je izredno odporno proti lezenju in oksidaciji. Prav tako sta zanj značilni dobra varivost in žilavost. V tej študiji smo proučevali lastnosti v povezavi z žilavostjo osnove P91 in zvarov. Prav tako je bila določena prehodna temperatura krhkega loma (DBTT - ductile-to-brittle transition temperature) osnovnega materiala P91. Mikrostruktura osnovnega materiala in zvarov vsebuje martenzit in popuščani martenzit ter nekatere precipitate. V preizkušu DBTT je bila ugotovljena žilavost jekla P91 v obliki osnovnega materiala in zvarov kar 80 J tudi pri -60 °C. Žilavost zvarov P91 meri pri sobni temperaturi 92 joulov.

**Ključne besede:** topotna odpornost jekla CrMo, varjenje, topotna obdelava

#### Abstract

P91 CrMo steel is extensively used at elevated temperature applications in fossil fire and nuclear power plants for tube and piping due to its excellent creep and oxidation resistance. Also they should have acceptable weldability and toughness. In this study microstructure and toughness properties of P91 base and weld metal were investigated. The ductile-brittle transition temperature (DBTT) of P91 base metal was also figured out. The microstructures of the base and weld metal have martensite and tempered martensite and some precipitates. In the (DBTT) test, toughness of P91 steel as base metal was observed as high as 80J even at -60 oC. Toughness of P91 weld metal has 92 Joule at room temperature.

**Keywords:** Heat resistance CrMo steels, Welding, Heat treatment

## 1 Uvod

Glavna težava v elektrarnah je že leta povezana z zmanjševanjem izpustov ogljikovega dioksida in energetsko učinkovitostjo. Kot odgovor na to težavo so bili razviti ultrasuperkritični (USC – ultra-supercritical) generatorji za elektrarne. Vendar pa je ta tehnologija privedla do še ostrejših pogojev, npr. višjih temperatur, prav tako pa je vplivala na merila glede lastnosti materialov, ki se uporabljajo v elektrarnah. 9-odstotno Cr-legirano feritno jeklo, kot je P91, ki je primerno za

## 1 Introduction

The first main problem in power plants for years has been reducing carbon dioxide emissions and energy efficiency. Ultra-super critical (USC) power plants generators have been developed as solution to this problem. But this technology has brought more severe conditions such as higher temperature and also has affected material properties criteria used in power plants. 9%Cr ferritic creep resistant alloy steels such as P91, capable of long term service up to 650°C- 700 °C, were used at USC plant applications such as the

dolgotrajno uporabo pri temperaturah do 650 °C–700 °C, se v sistemih USC v elektrarnah uporablja npr. za glavne parne vode, toplotne izmenjevalnike in cevovode za vroče tekočine, saj bolje prevaja toploto, se manj razteza ob povišanih temperaturah ter je bolj odporno proti nabrekanju kot avstenitno jeklo [1, 2].

Mikrostrukturo 9-odstotnih Cr-legiranih feritnih jekel sestavlja popuščani martenzit, utrjujejo pa jo dislokacije, topljenci in precipitati. Varjenje jeklenih cevi P91 poslabšajo z lezenjem povezane lastnosti, v varilnih materialih, podvrženih topotni obdelavi po varjenju (PWHT - post-weld heat treatment) z namenom popravil pa nastajajo mikrostrukturne napake. Vendar pa je višja ali nižja stopnja PWHT lahko nevarna, saj vodi višja temperatura PWHT v ogrobitev precipitatov, nižja temperatura PWHT pa v večja odstopanja s trdoto povezanih lastnosti [3].

Življenska doba jeklenih cevi se hitro krajša v povezavi z nastanjem razpok pri lezenju in njihovo rastjo. Kot je že bilo omenjeno, temperatura in čas topotne obdelave po varjenju učinkujeta na zvar in na topotno vplivana območja (HAZ - heat-affected zone). Ugotovljena so bila razpokana območja jeklenih cevi P91 in P92, predvsem na lokacijah TIPA IV [4]. Postopki PWHT so namenjeni izboljšanju žilavosti in voljnosti ter zmanjšanju trdnosti topotni vplivanih območij HAZ. Upravljanje časa in temperature PWHT bi lahko bilo izredno pomembno z vidika življenske dobe varjenih cevi. Številni raziskovalci priporočajo temperature PWHT 750 ali 760 °C za jekla P91 in P92. Raziskave PWHT in njenih učinkov so sprožile povečanje uporabe jeklenih cevi P92 [5, 6, 7, 8].

main steam pipe, heat exchangers and hot fluid pipes due to having the better thermal conductivity, lower thermal expansion and superior swelling resistance than austenitic steels [1, 2].

The microstructure of 9% Cr ferritic steels is tempered martensite and are hardened by dislocations, solutes and precipitates. Welding of P91 steel pipes aggravate the creep properties and the welding materials subject to PWHT to repair the microstructure defects. But lower or higher PWHT conditions could be hazardous because high PWHT temperature gives rise to more coarsening precipitates or lower PWHT temperature cause of larger differences in the hardness properties [3].

The total life of the steel pipes tend to be decreasing because of creep crack initiation or growth behavior. As mentions PWHT temperature or times effects the crack behaviours of weld and HAZ regions. Observed welding crack areas of P91 and P92 steel pipes condensed on TYPE IV location [4]. After the weld PWHT applications are improving the toughness and ductility and reduce the hardness of HAZ. Controlling of PWHT times or temperature could be extremely important regarding the life of welding pipes. Recommended PWHT temperatures are presented 750 or 760 °C for P91-P92 steels by many researches. Especially the increase of P92 steel pipes in the last 10 years has compelled the research of PWHT conditions and its effects [5, 6, 7, 8].

## 2 Experimental Procedure

In this work, P91 steel plate was used as base metal as received condition (tempered). P91 all weld metal was produced by shielded metal arc welding (SMAW) in Gedik Welding Company in Turkey as explained in detail

## 2 Poskusni postopek

V tej raziskavi je jeklena plošča P91 predstavljala osnovni material (poboljšan). Vsi zvari P91 so bili proizvedeni po postopku ročnega obločnega varjenja z oplaščeno elektrodo (SMAW - shielded metal arc welding) v podjetju Gedik Welding Company v Turčiji, kot je že bilo pojasnjeno v prejšnji študiji [9]. Preglednica 1 prikazuje kemijsko sestavo osnovnega materiala in zvarov P91.

Kot je že bilo omenjeno, je bil osnovni material P91 uporabljen v nespremenjenih pogojih. Vendar pa so bili vsi zvari P91 štiri (4) ure izpostavljeni topotni obdelavi po varjenju pri temperaturi 760 °C. Vzorci so bili jedkani z jedkalom vilella in metalografsko pregledani ter pripravljeni za mikroskopiranje z optičnim mikroskopom in vrstičnim elektronskim mikroskopom (SEM). Mikrostruktturni pregledi vseh vzorcev so bili izvedeni z optičnim mikroskopom Nikon MA 100 Eclipse. Analize z vrstičnim elektronskim mikroskopom so bile izvedene z elektronskim mikroskopom Zeiss Evo LS 10.

Vzorci za Charpyjev udarni preizkus so bili pripravljeni skladno s standardom TS EN ISO 14556 za osnovne materiale in zvare. Charpyjev udarni preizkus osnovnega materiala P91 je bil izveden pri -60, -50, -40, -30, -20, -10, 0 in 30 °C, zvarov P91 pa pri sobni temperaturi. Charpyjevi udarni preizkusi so bili izvedeni s preizkusno napravo zname Zwick/Roell

in the previous work [9]. Table 1 shows the chemical compositions of P91 base and weld metals.

As mentioned above, P91 base metal was characterized as received condition. However P91 all weld metal was subjected to the post weld heat treatment at 760 °C for four hours. Samples were etched by using vilella etchant for metallographic examinations and made for optical microscope and SEM characterization. Microstructural examination of all samples was performed by using a Nikon MA 100 Eclipse optical microscope. Scanning electron microscope (SEM) analyses were carried out by using a Zeiss Evo LS 10 model electron microscope.

Charpy impact test samples were prepared according to TS EN ISO 14556 standard from base and weld metals. While charpy impact tests of P91 base metal were performed at -60, -50, -40, -30, -20, -10, 0 and 30 °C, that of P91 weld metal was carried out at room temperature. Charpy impact tests were performed with a Zwick/Roell brand 450 Joule-capacity test device. After the charpy impact tests, fracture surfaces of the samples were investigated by scanning electron microscope (SEM).

## 3 Results and Discussion

Figure 1 shows respectively optical microscope photographs of P91 base and

**Preglednica 1:** Pregled kemijske sestave jekla in zvarov v študiji (wt. %)

**Table 1.** Chemical composition of the steels and weld metals examined in the present study (wt %)

Vzorec / Specimen	C	Si	Mn	P	S	Cr	Mo	Ni	V	Fe
Jeklo P91/ Steel P91	0,095	0,259	0,578	0,006	<0,0005	8,810	1,071	0,187	0,211	Bal.
Zvar P91 / weld metal P91	0,096	0,202	0,743	0,012	0,013	9,425	1,061	0,474	0,231	Bal.

s kapaciteto 450 joulov. Po Charpyjevih udarnih preizkusih so bile površine razpok vzorcev proučene z vrstičnim elektronskim mikroskopom (SEM).

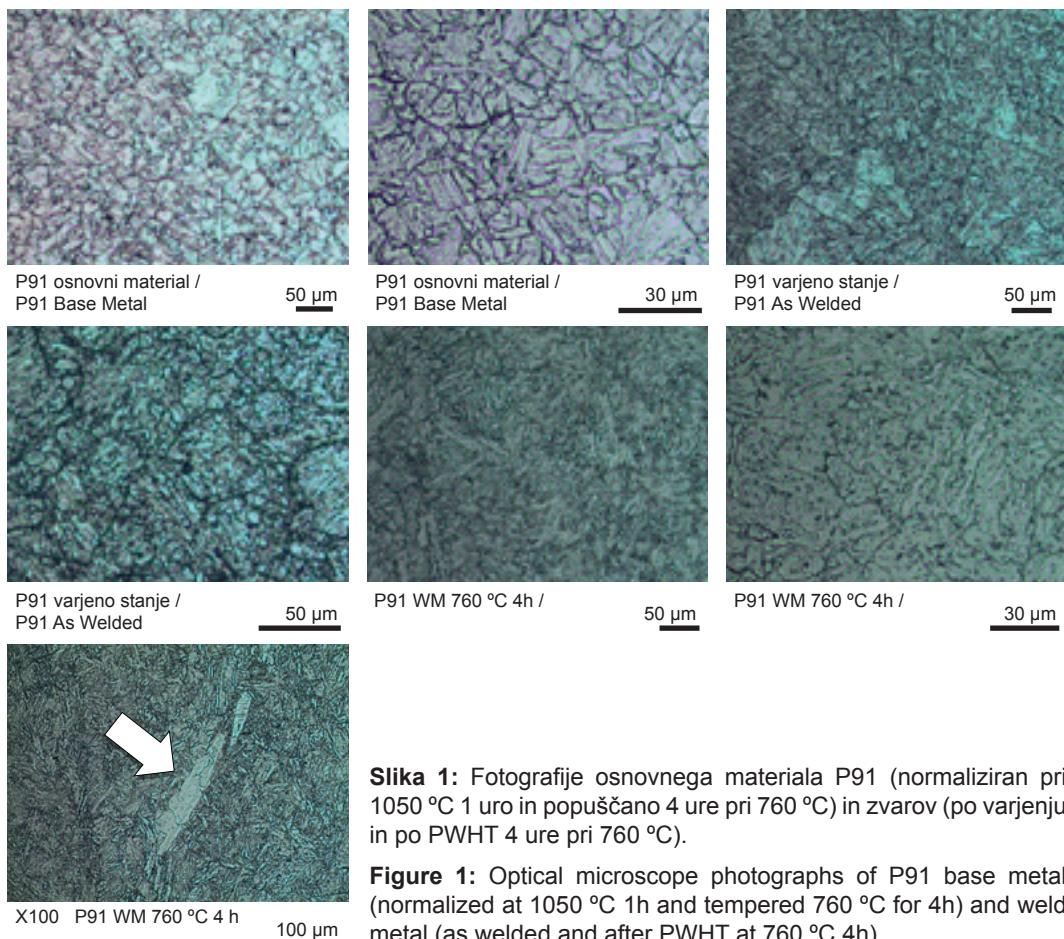
### 3 Rezultati in razprava

Na Sliki 1 sta prikazani mikroskopski fotografiji osnove P91 in zvarov po toplotni obdelavi po varjenju pri 760 °C v času 4 ur.

Kot prikazuje Slika 1, je bila tako pri temeljnem materialu kot zvarih P91 zaznana faza popuščanega martenzita. Morfologija

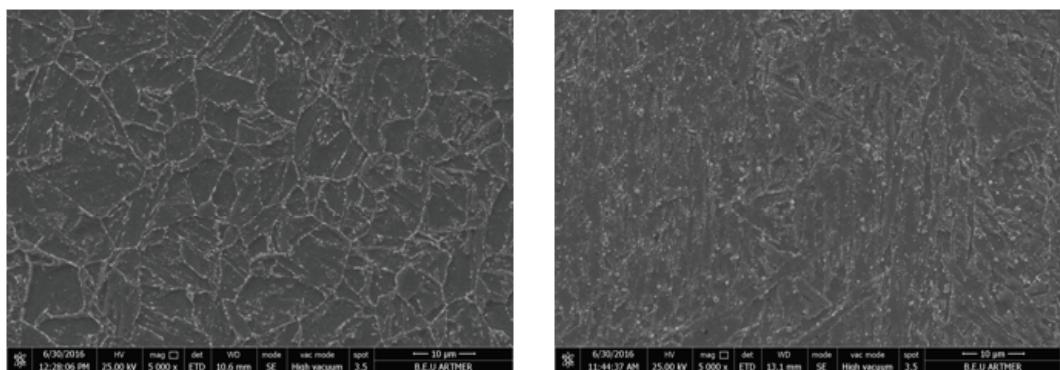
weld metals after post weld heat treatment at 760 °C for four hours.

As shown in Figure 1, it was observed that both of the P91 base and weld metals include tempered martensite phases. In the weld metal, the martensite phase has columnar and/or lath morphology. SEM examination was performed to determine the distribution of carbides, which were present in samples within the structure. Figure 2 shows the carbides as precipitates in both of P91 base and weld metal.



**Slika 1:** Fotografije osnovnega materiala P91 (normaliziran pri 1050 °C 1 uro in popuščano 4 ure pri 760 °C) in zvarov (po varjenju in po PWHT 4 ure pri 760 °C).

**Figure 1:** Optical microscope photographs of P91 base metal (normalized at 1050 °C 1 h and tempered 760 °C for 4h) and weld metal (as welded and after PWHT at 760 °C 4h).



**Slika 2:** Mikrostruktura a) osnovnega materiala P91 in b) zvarov

**Figure 2:** The microstructure of a) P91 base metal and b) weld metal

martenzitne faze v zvarih je bila in stebričasta in/ali letvasta. Izvedena je bila analiza z vrstičnim elektronskim mikroskopom z namenom opredelitev distribucije karbidov, prisotnih znotraj struktur vzorcev. Slika 2 prikazuje karbide v obliki precipitatov tako v osnovnem materialu kot zvarih P91.

Precipitati (bele kroglaste strukture) so porazdeljeni znotraj strukture. Prav tako je bilo ugotovljeno, da karbidi nastajajo pretežno na robovih obstoječih avstenitnih zrn. Sklepati je mogoče, da so majhne količine kroglastih karbidov porazdeljene znotraj matrice. Precipitati verjetno vključujejo M23C<sub>6</sub>, M7C<sub>3</sub>, M2C in karbide MX. V zvaru je bila zaznana tudi faza delta ferita. Delta ferit nastaja v primeru neuravnoveženih elementov, ki tvorijo ferit/avstenit, in varičnih parametrov, še posebej dovajane topote, ki učinkuje na nastanek delta ferita v zvarih jekla z visoko vsebnostjo Cr-Mo [10–12].

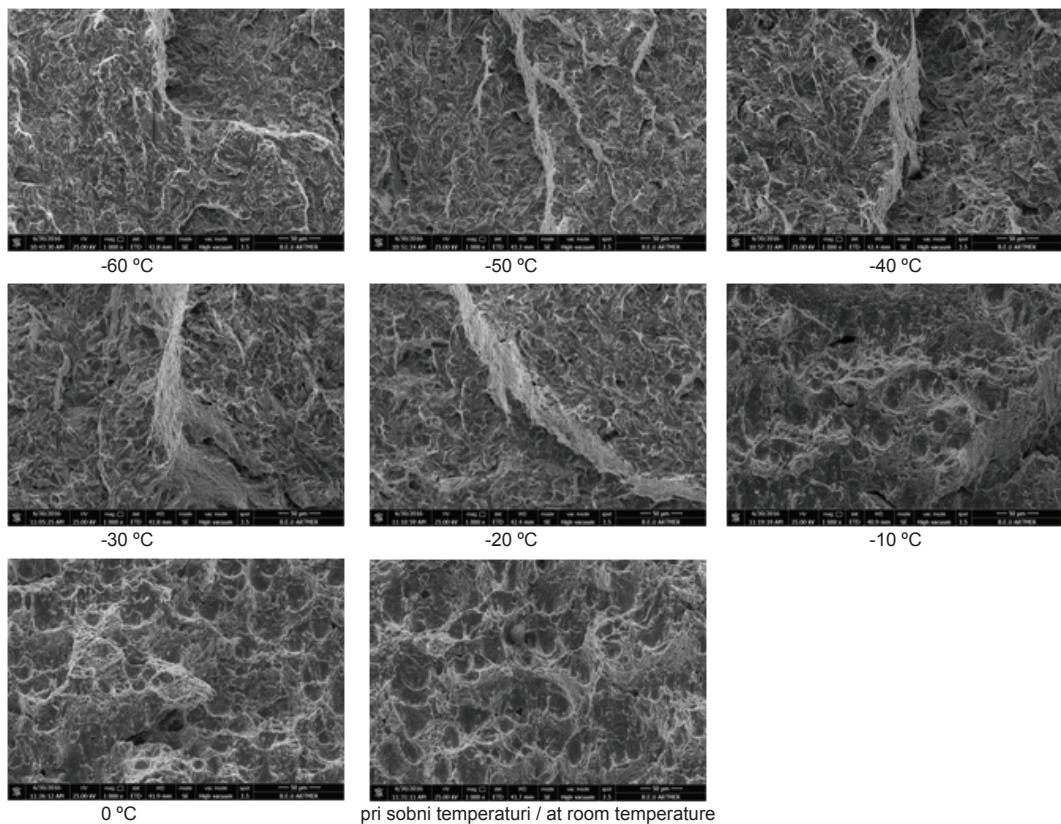
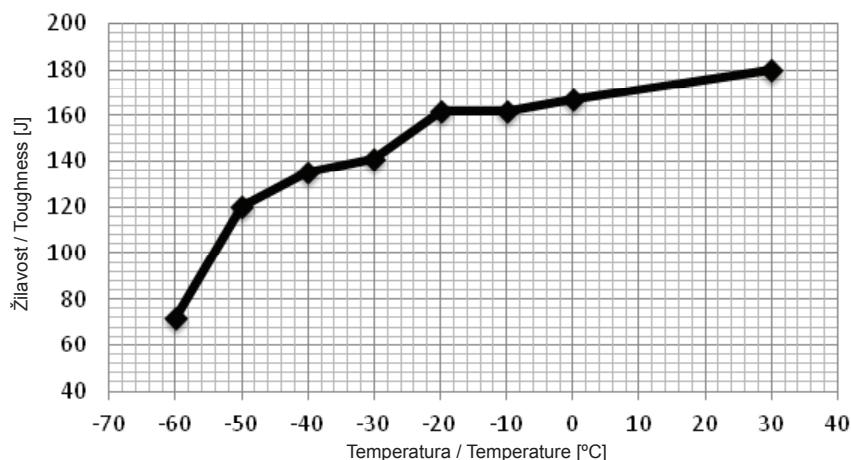
Na Sliki 3 je prikazana prehodna temperatura krhkega zloma osnovnega materiala P91 ter žilavost zvara P91. Na Sliki 3 je prikazano, da so z osnovnim materialom P91 povezane višje vrednosti žilavosti tudi pri nizkih temperaturah, npr. -60 °C. Vendar pa žilavost zvarov P91 pri

The precipitates seem to be distributed (white spherical structures) within the structure. It was also observed that the formation of carbides was generally in prior austenite grain boundaries. Additionally, it can be assumed that spherical carbides in small quantities showed a distribution within the matrix. The precipitates are probably M23C<sub>6</sub>, M7C<sub>3</sub>, M2C, and MX carbides. Delta ferrite phase was also detected in the weld metals. Delta ferrite occurs in case of unbalanced ferrite/austenite-forming elements and welding parameters, especially heat input, are important for formation of delta ferrite in the weld metal of the high Cr-Mo steel weld metal [10–12].

Figure 3 shows DBTT of P91 base metal and toughness value of P91 weld metal. As can be seen in Figure 3, P91 base metal has high toughness values even at low temperature such as -60 °C. However, the toughness of P91 weld metal did not reach that of the P91 base metal at room temperature. It can be assumed that the lower degree of the toughness values in the weld metal results from the delta ferrite phase, the inhomogeneous distribution of the carbides, the martensite lath structure and finally the oxide particles in the weld metal.

**Slika 3.**  
Prehodna  
temperatura  
krhkega zloma  
osnovnega  
materiala P91

**Figure 3.** DBTT of  
P91 base metal



**Slika 4:** Posneti razpokanih površin osnovnega materiala P91 pri različnih temperaturah, zajeti z vrstičnim elektronskim mikroskopom

**Figure 4.** SEM photographs of fractured surfaces of P91 base metal at different temperatures

sobni temperaturi ne dosega vrednosti osnovnega materiala P91. Sklepati je mogoče, da je nižja stopnja žilavosti zvarov posledica faze delta ferita, nehomogene porazdelitve karbidov, letvaste strukture martenzita in oksidnih delcev v zvarih.

Na Sliki 4 so prikazani posnetki razpokane površine osnovnega materiala in zvarov po Charpyjevih udarnih preizkusih, zajeti z vrstičnim elektronskim mikroskopom.

#### 4 Sklepi

V tej študiji smo proučevali lastnosti v povezavi z žilavostjo osnove P91 in zvarov. Rezultati študije so prikazani spodaj.

1. V zvarih lahko nastaja delta ferit.
2. Karbidi so nastajali na predhodnih robovih avstenitov in robovih letvastega martenzita ter so bili porazdeljeni znotraj matrice tako osnovnega materiala kot zvarov.
3. Žilavost jekla P91 v obliki osnovnega materiala je merila kar 80 J tudi pri temperaturi -60 °C.
4. Žilavost jekla P91 v obliki zvarov je bila pri sobni temperaturi sprejemljiva.

#### 5 Zahvale

To študijo je v sklopu projektov SAN-TEZ pod številko vloge 0374.STZ.2013-2 podprtlo turško Ministrstvo za znanost, industrijo in tehnologijo.

Figure 4 shows SEM images of the fractured surface of base and weld metals after Charpy-V impact tests.

#### 4 Conclusion

In this work, toughness properties of P91 base and weld metal were investigated. The results of the study are given below.

1. Delta ferrite can form in the weld metal.
2. The carbides formed in the prior austenite boundaries and lath martensite boundaries and also distributed within the matrix of the base and weld metals.
3. The toughness of P91 steel as base metal was observed as high as 80J even at -60°C.
4. The toughness of P91 weld metal has above acceptable value at room temperature.

#### 5 Acknowledgements

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