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CENTRELINE FORMATION OF THE Nb(C,N) EUTECTIC IN 0.15 % C; 0.0071 % N; 0.022 % Nb; 0.033 % Al AND 0.003 % S STRUCTURAL STEEL

SREDINSKO IZCEJANJE IN NASTANEK EVTEKTIKA Nb(C,N) V KONSTRUKCIJSKEM JEKLU Z 0,15 % C; 0,0071 % N; 0,022 % Nb; 0,033 % Al IN 0,003 % S

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During a routine control, a very small through thickness reduction of area was found for one tensile specimen of a 90-mm plate. Careful investigations of the fracture and the section of specimens cut from the as-solidified continuously cast 250-mm slab showed that the cause was the presence of coarse particles of niobium carbo-nitride as a constituent of the quasi-eutectic Fe-Nb(C,N) that forms because of the centerline segregation of niobium.

Key words: structural steel, heavy plates, reduction of area, eutectic niobium carbo-nitride

Pri rutinski kontroli lastnosti jekla je imel raztržni preizkušanec 90-milimetrske mm plošče zelo majhno kontrakcijo v smeri debeline. Preiskava prelomne površine in prereza preizkušancev, izrezanih iz kontinurino litega 250-milimetrskega slaba, je pokazala, da je vzrok zanjo prisotnost velikih zrn niobijevega karbonitrida v spačenem evtektiku Fe-Nb(C,N), ki je nastal zaradi sredinske segregacije niobija.

Ključne besede: konstrukcijsko jeklo, debele plošče, kontrakcija v smeri debeline, evtektik niobijevega karbonitrida

1 INTRODUCTION

The reduction of area in the through thickness direction is an essential mechanical property of thick steel heavy plates intended for fillet welds. In the standard EN 10164[1] three quality classes, Z15, Z25 and Z35, with minimal average values for the through thickness reduction of area of three tests, 15 %, 25 % and 35 %, and minimal individual values, 10 %, 15 % and 25 %, respectively, are specified. During routine testing in a mechanical laboratory for one specimen only 9.5 % of the through thickness reduction of area was found, although the declared plate class was Z35. The sample is shown in **Figure 1**. According to Vodopivec et al. [2] the content of sulphur is the primary reason for the low ductility in the through thickness direction because of the lamelar tearing with fracture propagation also along the interface between the sulphide inclusion and the ferrite matrix. The mass fraction of sulphur in the tested steel, which was only 0.003 %, excludes the possibility of a low reduction of area due to sulphide inclusions.

Figure 1: Macroscopic image of S 355 J2+N structural steel, showing the reduction of area specimen taken from a 90-mm heavy plate in the thickness direction

Slika 1: Makroskopski posnetek kontrakcijskega preizkušanca konstrukcijskega jekla S 355 J2+N. Preizkušanec je vzet po debelini iz 90-milimetrske debele plošče

A small amount of niobium was added to the investigated structural steel to achieve the required mechanical properties. The addition of Nb could also affect the through thickness ductility of heavy plates because of the formation of coarse niobium carbo-nitride particles as constituents of the degenerated eutectic Fe-Nb(C,N), which may form with a high content of niobium or because of defective solidification of the steel[3].

To identify the cause of the low reduction of area, detailed investigations of specimens cut at different distances from the surface of the as-solidified slab were carried out.

2 EXPERIMENTAL

The structural steel (S 355 J2+N) was melted in an EAF (electric arc furnace), VD (vacuum degassing) treated, continuously cast and cut into slabs of dimensions (250 × 1085 × 4770) mm. The slabs were cooled to

Table 1: Chemical composition of the S 355 J2+N steel grade in mass fractions *w*/%
Tabela 1: Kemijska sestava jekla S 355 J2+N v masnih deležih *w*/%

Element	C	Si	Mn	P	S	Cr	Cu	Ni	Al	Nb	Ti	N
w/%	0.15	0.49	1.10	0.018	0.003	0.14	0.29	0.12	0.033	0.022	0.005	0.0071

room temperature and after surface grinding reheated in a pusher-type furnace to a temperature of 1250 °C and hot rolled to 90-mm-thick plates. The chemical composition of the heat is listed in **Table 1**. First, samples perpendicular to the slab casting direction were examined after grinding and deep-etching for 40 min in 25 % H_2SO_4 at 70 °C, which revealed the as-cast macrostructure. From this specimen, samples 1, 2 and 3 in the thickness direction were cut out for metallographic examination, as shown in **Figure 2**. From the 90-mm heavy plate, specimens were cut out in the thickness direction and submitted for tensile testing and examinations with optical and scanning electron microscopes (SEM) as well as energy-dispersive X-ray spectroscopy (EDXS).

3 RESULTS AND DISCUSSION

Figure 3 shows a secondary-electron image of a fracture surface of one specimen with coarse niobium carbo-nitride inclusions and small MnS inclusions. The spots of the EDXS analyses of both inclusions are marked with arrows in **Figure 3** and the results are given in **Table 2**. In the mapping micrographs in **Figure 4** the bright areas represent the element in the particles and show the morphology of the particles and the main elements in large inclusions. Most of the particles observed on the fracture surface showed a large content of niobium. On the basis of fractographs it was concluded that niobium-containing particles (Nb,Ti)(C,N) were the main cause for the poor through thickness reduction of area of the steel plate.

From the location of the fracture of the tensile specimen shown in **Figure 1,** we assumed that the source of the coarse precipitates was a very strong centreline segregation during the solidification of the steel slab.

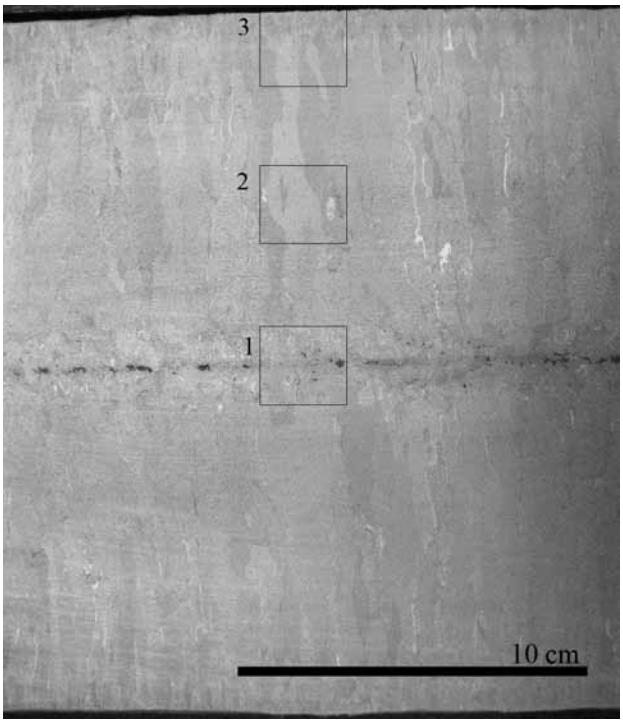

Figure 2: As-cast sample taken from slab perpendicular to the casting direction
Slika 2: Vzorec iz slaba pravokotno na smer ulivanja jekla

Table 2: Results of the spot EDXS analyses. The place of the analysis is marked with an arrow in **Figure 3**.
Tabela 2: Rezultati točkovne EDXS analize. Mesto analize na **sliki 3** je označeno s puščico.

	Element	Fe	Mn	S	Ti	Nb
conc. *w*/%	(Nb,Ti)(C,N)	28.661	0.728	–	3.853	66.758
	MnS	3.747	70.306	25.861	–	–

Tabela 3: Results of the spot EDXS analyses. The place of the analysis is marked with an arrow in **Figure 5**.
Table 3: Rezultati točkovne EDXS analize. Mesto analize na **sliki 5** je označeno s puščico.

	Element	C	Fe	Mn	S	Ti	Nb	Al	Pb
conc. *w*/%	(Nb,Ti)(C,N)	3.18	33.09	–	–	3.76	59.97	–	–
	MnS	–	3.56	61.89	33.09	–	–	–	–
	Pb	2.01	9.16	–	–	–	–	1.62	84.46

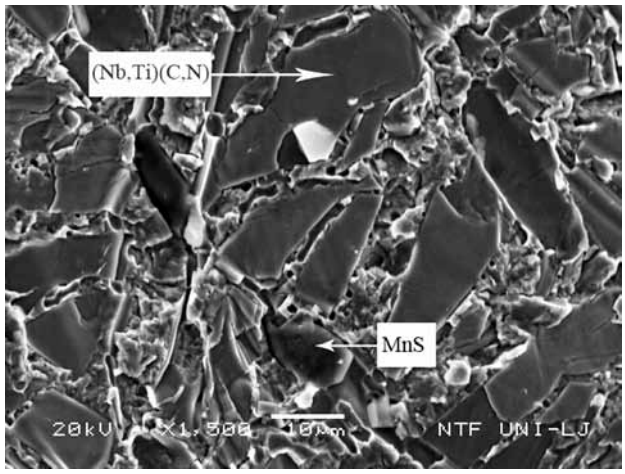

Figure 3: SEM fractograph (secondary electron image) of the reduction of area specimen. The analysed particles are marked with arrows, and the EDXS analyses are presented in **Table 2**
Slika 3: SEM-slika (sekundarni elektroni) prelomne površine kontrakcijskega preizkušanca. Točke opravljene EDXS analize prikazujeta puščici, rezultati so podani v **tabeli 2**

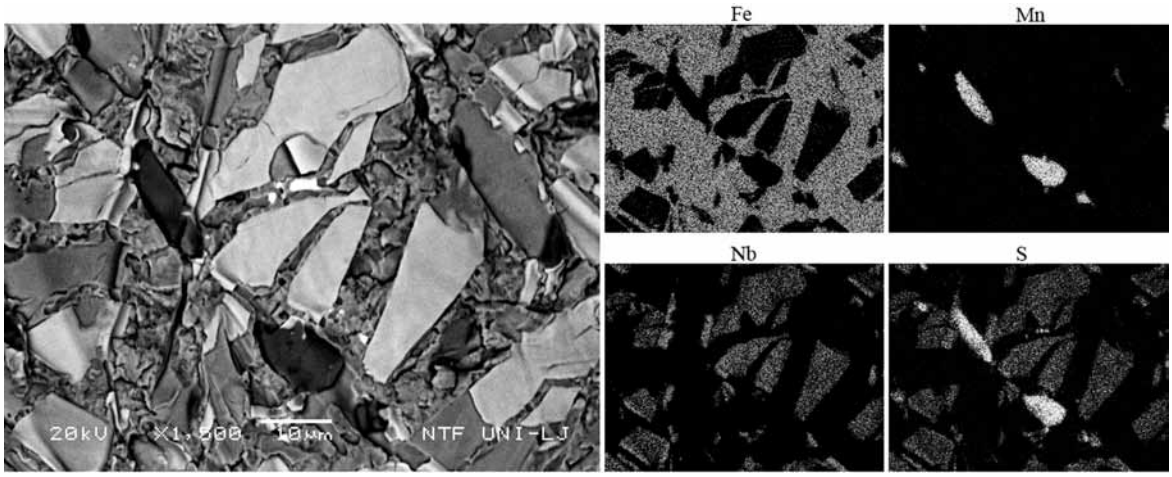

Figure 4: EPMA mapping of Nb(C,N) and MnS particles
Slika 4: Ploskovna mikroanaliza delcev Nb(C,N) in MnS

This conclusion is confirmed by the fact that from three samples, as shown in **Figure 2,** the niobium-rich precipitates were only found in the specimen cut from the slab centre in sample number 1. The precipitates are very similar to the Fe-Nb(C,N) eutectic known as "Chinese script"[4]. Besides the niobium-rich particles, a minor number of very small manganese sulphide inclusions and lead droplets were found. All these phases were only found in the centreline of the cast slab. The results of the spot EDXS analyses from the cast slab are presented in **Table 3** and the spots of the analyses are marked in **Figure 5**. The analyses show that the niobium carbo-nitride particles also contain the mass fraction of Ti approximately 3.7 %, despite there being only 0.005 % of titanium in the steel originating from the steel scrap used. A similar composition of niobium carbo-nitride was reported for Nb-Ti micro-alloyed steels[5].

The solubility of the niobium carbo-nitride with the approximate composition $Nb(C_{0.9}N_{0.1})$ in structural steel is given by the equation[6,7]:

$$\lg\left[(w(\mathrm{Nb})w(\mathrm{C})+\frac{12w(\mathrm{N})}{14}\right]=2{,}26-\frac{6770}{T}$$

with w(Nb), w(C), and w(N) being the mass fractions of the elements in the steel and T being the temperature in K.

Considering the actual contents of niobium, carbon and nitrogen, a solution temperature of 1140 °C was deduced, indicating that the slab soaking temperature was sufficient for a complete solution in austenite of the niobium carbo-nitride with the approximate composition $Nb(C_{0.9}N_{0.1})$. The fact that coarse niobium-rich precipitates were also found in the hot-rolled plate after heating the slabs to 1250 °C indicates that their composition differs from that of the soluble niobium carbo-nitride. The solubility of niobium carbide in austenite is greater than the solubility of niobium nitride[8,9]. It is assumed that the stability of particles in the investigated steel is due to their high content of nitrogen.

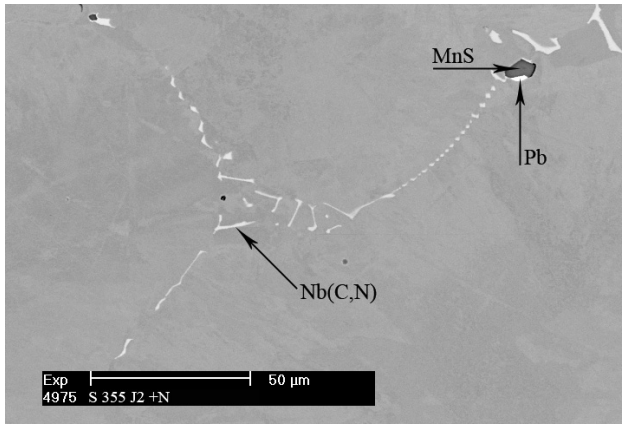

Figure 5: SEM picture of degenerated eutectic in form of "Chinese script" from the sample number 1 of the as-cast slab. The analysed particles are marked with arrows, and the EDXS analyses are presented in **Table 3**
Slika 5: SEM-posnetek spačenega evtektika z obliko "kitajske pisave" iz vzorca številka 1 kontinuirno ulitega slaba. Točke opravljene EDXS-analize prikazujejo puščice, rezultati so podani v **tabeli 3**

The shape and size of the coarse carbo-nitride particles suggest that they are constituents of a degenerated quasi-eutectic Fe-Nb(C,N). The location of the eutectic in the centre of the slab and the composition of the steel suggest that its formation is an improper solidification process related to a high casting temperature, a high slab solidification rate or a deficiency in the secondary slab cooling.

4 CONCLUSIONS

When considering the contents of carbon, nitrogen and niobium in a steel, all the carbo-nitride phase with the approximate composition $Nb(C_{0.9}N_{0.1})$ is in a solid solution in austenite at 1140 °C. Since the slab soaking temperature was 1250 °C, it is evident that the carbo-nitride found in the examined steel does not have the quoted composition and that it has a higher content of nitrogen and correspondingly a higher solution tempera-

ture in austenite. The shape and the size of the niobium-rich particles suggest that they are the constituents of a degenerated eutectic Fe-Nb(CN) that formed because of the improper solidification process of continuous cast slabs.

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