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THE INFLUENCE OF THE MICROALLOYING ELEMENTS OF HSLA STEEL ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES

VPLIV MIKROLEGIRNIH ELEMENTOV NA MIKROSTRUKTURO IN MEHANSKE LASTNOSTI HSLA JEKLA

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In this work, hot-forged, high-strength, low-alloy (HSLA) steels based on the chemical composition 34CrNiMo6 were investigated in order to determine the type of precipitates forming and their effect on the mechanical properties. The individual steels were microalloyed with one of the following elements or a combination of elements: niobium, titanium or zirconium and vanadium. The compositional changes in the microstructure and the various kinds of precipitates observed in the microalloyed steels were examined using energy-dispersive spectroscopy (EDS). Optical and scanning electron microscopy studies revealed that the addition of microalloying elements did not change very much the main microstructural features due to the fact that all the microstructures consisted of tempered martensite and finely dispersed carbide precipitates along martensite laths. In all the microstructures relatively large precipitates of the microalloyed elements were observed. This could be a possible reason for observing no significant improvement in the mechanical properties.

Keywords: high-strength, low-alloy (HSLA) steels, microalloying elements, precipitates, mechanical properties, electron microscopy

Vroče kovano jeklo HSLA kemijske sestave 34CrNiMo6 smo raziskovali z namenom, da bi določili vrsto precipitatov, ki se tvorijo, in njihov vpliv na mehanske lastnosti. Posamezna jekla so bila mikrolegirana z enim od naslednih elementov ali s kombinacijo le-teh: z niobijem, s titanom ter cirkonijem in z vanadijem. Mikrostrukturne spremembe in različne vrste precipitatov smo študirali z metodo EDS. Preiskave s svetlobnim in vrstičnim elektronskim mikroskopom so potrdile, da dodatek mikrolegirnih elementov ni bistveno spremenil osnovne mikrostrukture, saj so bile vse mikrostrukture iz popuščenega martenzita z izločenimi karbidnimi precipitati po martenzitnih letvah. Pri vseh mikrostrukturah so bili opaženi tudi precej veliki precipitati mikrolegirnih elementov, ki so po vsej verjetnosti razlog za to, da se mehanske lastnosti niso bistveno izboljšale.

Ključne besede: visokotrdna malo legirana jekla (HSLA), mikrolegirni elementi, precipitati, mehanske lastnosti, elektronska mikroskopija

1 INTRODUCTION

Microalloyed, high-strength, low-alloy (HSLA) steels are technologically important structural materials.[1,2] These steels contain small amounts of alloying elements, such as titanium, niobium, vanadium or zirconium, which enhance the strength through the formation of stable carbides, nitrides or carbonitrides. Titanium also readily forms stable nitrides at high temperatures and is used to control the nitrogen content in the alloys. The microalloying elements niobium and vanadium are added to microalloyed cast steels primarily to provide grain refinement and a response to aging. Niobium, often at levels of less than 0.05 %, effectively prevents undesirable grain growth and can also contribute to precipitation strengthening. Vanadium, in particular at levels of less than 0.1 %, forms strengthening carbonitride precipitates.[3,4] Precipitation hardening by fine carbonitride particles has been used for many years to increase the strength of microalloyed steels. During the thermomechanical processing, carbonitrides may nucleate in austenite during forging, at the γ/α interface during transformation (interphase precipitation), or in supersaturated ferrite during the final cooling.[5]

On adding such elements to steels with 0.008–0.03 % C and up to w = 1.5 % Mn, it became possible to produce fine-grained material with yield strengths between 450 MPa and 550 MPa, and with ductile/brittle transition temperatures as low as –70 °C.[6]

Nowadays, microalloyed cast steels have found many applications in the manufacturing of industrial parts, such as offshore platform nodes, centrifugal cast pipes, machinery supports, ingot moulds and buckets, which all used to be produced by expensive manufacturing processes.[7–9]

2 EXPERIMENTAL WORK

Hot-forged HSLA steels based on the chemical composition 34CrNiMo6 were investigated in order to determine the type of precipitates forming and their effect on the mechanical properties. Individual steels were microalloyed with one of the following elements or combination of elements: niobium (steel A), titanium

Table 1: Chemical compositions of the investigated steels microalloyed with niobium (steel A), titanium (steel B), vanadium and zirconium (steel C) and non-microalloyed (steel D) in mass fractions

Tabela 1: Kemijska sestava jekla, mikrolegiranega z niobijem (jeklo A), s titanom (jeklo B), z vanadijem in s cirkonijem (jeklo C) in nelegiranega jekla (jeklo D) v masnih deležih (%)

Steel	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Al	Nb
A	0.35	0.56	0.26	0.016	0.004	1.52	1.55	0.13	0.17	0.029	0.039

Steel	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Al	Ti
B	0.32	0.55	0.27	0.022	0.002	1.58	1.56	0.15	0.18	0.020	0.027

Steel	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	V	Al	Zr
C	0.34	0.54	0.27	0.017	0.002	1.58	1.55	0.21	0.23	0.030	0.031	0.030

Steel	C	Mn	Si	P	S	Cr	Ni	Mo	Al
D	0.34	0.60	0.26	0.007	0.012	1.55	1.58	0.20	0.030

(steel B) or zirconium and vanadium (steel C). For comparison a non-microalloyed steel (steel D) was chosen. The chemical compositions of the steels, as specified by the manufacturer, are given in **Table 1**.

Metallographic samples were prepared using standard polishing techniques and were then etched with 2 % Nital. The microstructure was examined in an optical microscope, a Nikon Microphot FXA. The carbide precipitates were investigated using a field-emission scanning electron microscope, a JEOL JSM 6500F, equipped with an energy-dispersive spectroscopy (EDS) analyzer (INCA X-SIGHT LN2 type detector, INCA ENERGY 450 software). The accelerating voltage during the EDS was 15 kV, the working distance 10 mm and the probe current 0.05 nA.

To evaluate the mechanical properties tensile tests were conducted on a 500-kN tensile machine, an Instron 1255.

3 RESULTS AND DISCUSSION

3.1 Microstructural characterization

The solubility data imply that in a microalloyed steel, carbides and carbonitrides of Nb, Ti, Zr and V will precipitate. While the primary effect of these fine dispersions is to control the grain size, dispersion strengthening will take place. The strengthening arising from this cause will depend both on the particle size and the interparticle spacing, which is determined by the volume fraction of the precipitate. These parameters will depend primarily on the type of compound that is precipitating, and this is determined by the microalloying content of the steel.[6]

In modern microalloyed steels there are at least three strengthening mechanisms that contribute to the final strength achieved. In the first mechanism, the precipitation takes place in the austenite and further precipitation occurs during the transformation to the ferrite. The precipitation of niobium, titanium and vanadium carbides has been shown to take place progressively as the interphase boundaries move through the steel. This is the second mechanism, called interphase precipitation. As this precipitation is normally on an extremely fine scale, occurring between 850 °C and 650 °C, it is likely to be the major contribution to the dispersion strengthening. If the rate of cooling through the transformation is high, leading to the formation of supersaturated plates of ferrite, the carbides will tend to precipitate within the grains, usually onto the dislocations, which are numerous in this type of ferrite. This is the third strengthening mechanism.[6]

Optical microscopy of the investigated steels revealed that the addition of different microalloying elements did not considerably change the main microstructural features due to the fact that all the microstructures consisted of tempered martensite and finely dispersed carbide precipitates along the martensite laths. In all the microstructures relatively large precipitates (up to 10 μm) of microalloyed elements were observed. After etching, in the optical microscope these precipitates can

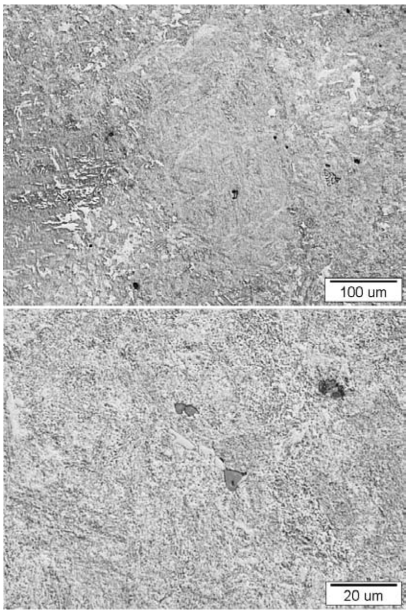

Figure 1: Optical micrographs of the steel microalloyed with niobium (steel A) at different magnifications

Slika 1: Mikrostruktura jekla, mikrolegiranega z niobijem (jeklo A), pri različnih povečavah (svetlobni mikroskop)

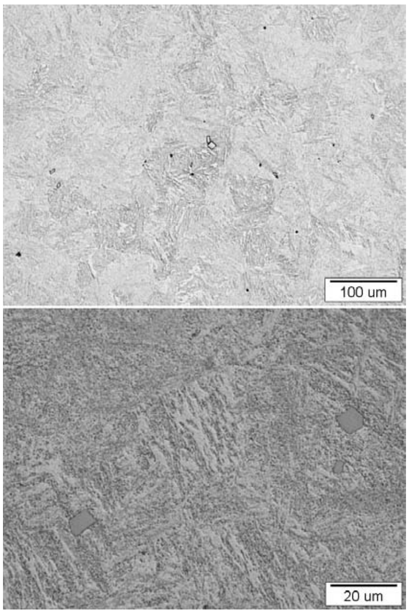

Figure 2: Optical micrographs of the steel microalloyed with titanium (steel B) at different magnifications

Slika 2: Mikrostruktura jekla, mikrolegiranega s titanom (jeklo B), pri različnih povečavah (svetlobni mikroskop)

be seen as typically coloured (brown-reddish for the niobium and titanium precipitates and yellow for the zirconium precipitates). Representative optical micrographs of the microalloyed steels are presented in **Figures 1, 2 and 3**.

In order to observe the microstructure more closely, the precipitates of the microalloying elements were investigated using scanning electron microscopy. In the steel A, the precipitates are very similar to the degenerated eutectic Fe-Nb(C, N) known as "Chinese script"[10,11] (**Figure 4**) that formed because of the improper solidification process of the cast ingots.

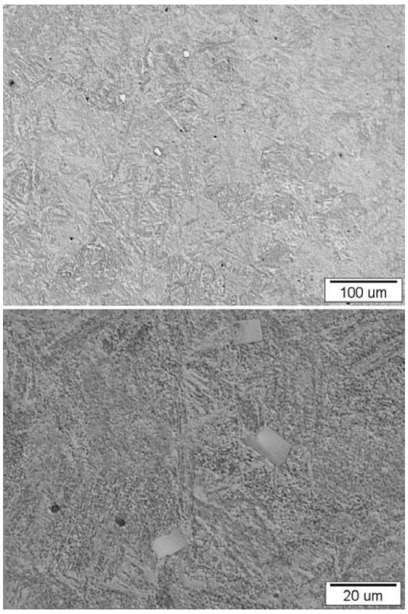

Figure 3: Optical micrographs of the steel microalloyed with vanadium and zirconium (steel C) at different magnifications

Slika 3: Mikrostruktura jekla, mikrolegiranega z vanadijem in cirkonijem (jeklo C), pri različnih povečavah (svetlobni mikroskop)

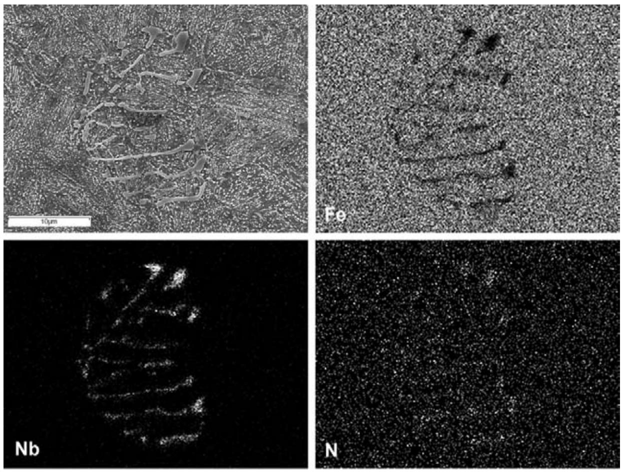

Figure 4: SE image and X-ray maps of niobium precipitate (steel A) in the form of "Chinese script"

Slika 4: SE-posnetek in porazdelitev elementov niobijevega precipitata v obliki kitajske pisave, določena z EDS

Figure 5 shows the large titanium nitride in the steel B, bounded to a complex, non-metallic inclusion composed of manganese sulphide and aluminium oxide.

A similar microstructure was also observed in the steel C, where large zirconium carbonitrides were found (**Figure 6**). EDS analyses of all three particles were performed and the chemical composition is listed in **Table 2**. Typical for the Nb, Ti and Zr carbonitrides is a regular geometrical shape, which is seen as a triangular, quadrilateral shape. Their shape is related to their crystal structure and is influenced by growing along the energetically most favourable crystal planes. Such particles are not desirable in the material because they are very hard and have sharp edges, which can cause a lowering of the mechanical properties of the material.

The aim of microalloying the steel is to obtain very fine Nb, Ti and Zr precipitates on the nanometre scale, which improves the material properties with relatively low costs. In our study it was found that the benefit of microalloying was suppressed by a non-optimised alloying process. We assumed that most of the microalloying elements were extracted from the melt as large particles instead of a finely dispersed precipitate.

Carbonitride precipitation has been the subject of many investigations that have studied the different aspects of the precipitates and their effects on the mechanical properties.[7] Based on these studies, the effective carbonitrides in strengthening are those formed in a fine form with an adequate distribution. As a result, the increase observed in the strength can be attributed to the precipitation of fine-scale carbonitrides in the form of interphase and random precipitates.

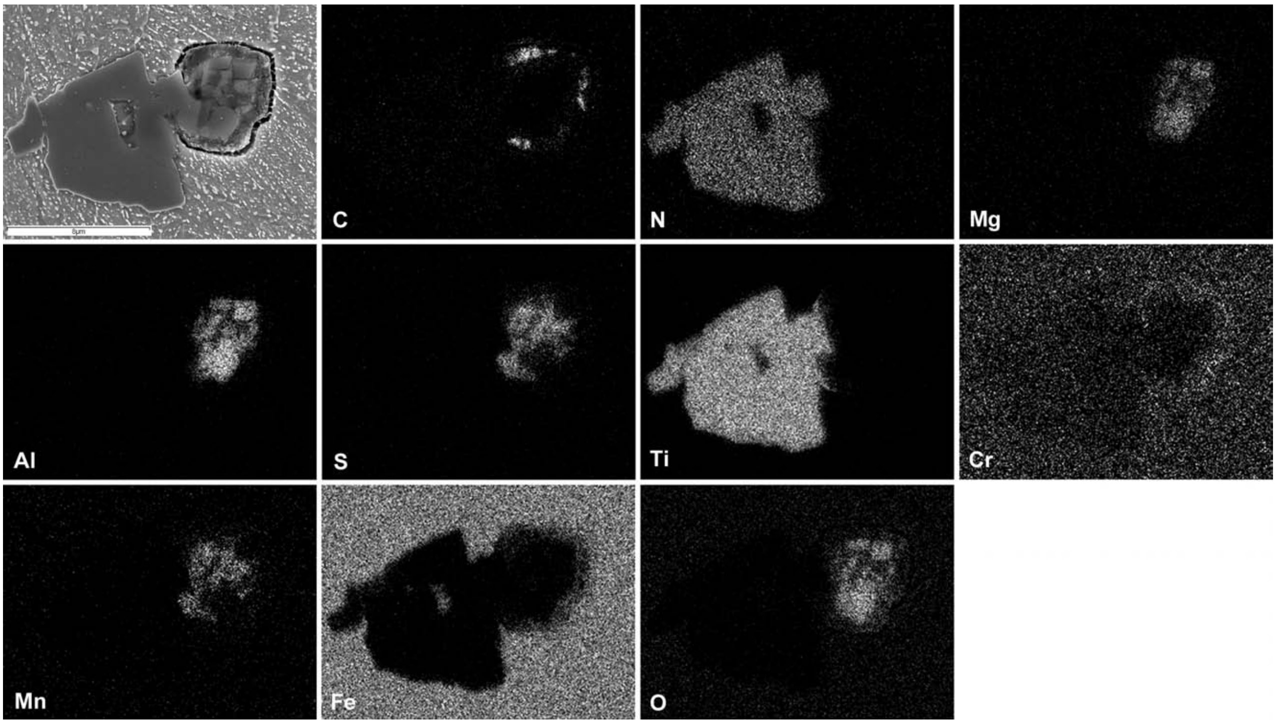

Figure 5: SE image and X-ray maps of the titanium precipitate in the steel B
Slika 5: SE-posnetek in porazdelitev elementov titanovega precipitata v jeklu B

3.2 Mechanical properties

The results obtained from the tensile tests are reported in **Table 3**. The properties reported in this table are the average properties of two testing specimens. According to these results, the presence of different microalloying elements does not significantly change the mechanical properties. It is possible that the benefit of the fine precipitates is eliminated with the presence of large Nb, Ti and Zr particles.

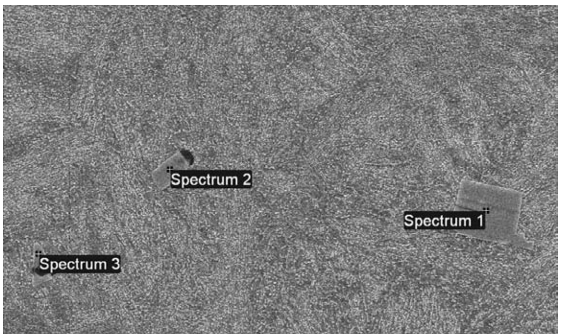

Figure 6: SE image of the zirconium precipitates in steel C
Slika 6: SE-posnetek cirkonijevih precipitatov v jeklu C

Table 2: Chemical composition for the analysed spectra in mass fractions
Tabela 2: Kemijska sestava analiziranih spektov v masnih deležih (%)

	C	N	Ti	Cr	Mn	Fe	Ni	Zr
Spectrum 1	10.80	11.50	0.31		0.61	6.75		70.03
Spectrum 2	15.32	9.22	0.40			7.20		67.86
Spectrum 3	10.85	6.53	0.81	0.83	0.75	31.49	0.47	48.26

Table 3: Mechanical properties of the investigated steels
Tabela 3: Mehanske lastnosti preizkušanih jekel

Steel	R_e /MPa	R_m /MPa	*A*5/%	*Z*/ %
A	611	805.5	18.6	53.1
B	637	806.5	18.7	64.6
C	627	808.5	19.2	65.2
D	619	786	21.1	62.2

4 CONCLUSIONS

The influence of the microalloying elements in HSLA steels on the microstructure and mechanical properties was investigated. It was found that Nb, Ti and Zr precipitate not only as fine particles but also as large particles with sharp edges. Based on the mechanical properties and the SEM/EDS analysis it is supposed that the benefit of microalloying elements is negated by these large particles and the results are almost the same mechanical properties as in the case of the non-microalloyed steel (steel D).

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