Walerij Kuzniecow, Konstantin Szapowałow

Effect of Nano-oxides on the Structure and Properties of Low-alloy Steel Weld Metal

Abstract: The article presents the effect of nano-oxides on the distribution of non-metallic inclusions as well as on the weld metal structure during welding of low-alloy steels. It has been ascertained that providing the weld pool with a 0.5% vol. of aluminium and titanium nano-oxides leads to the formation of acicular ferrite structure characterised by high mechanical properties.

Keywords: nano-oxides, low-alloy steels, aluminium, titanium

Introduction

The possibilities of increasing the level of mechanical characteristics of alloy and low-alloy welded joints by selecting appropriate welding materials and structural heat processing conditions are close to exhaustion. Progress in solving this issue can be achieved by using new methods for weld metal structure control applying the effects of influencing the strength and plasticity characteristics of the basic hardening factors such as the grain size, morphology, dispersion degree and volume fraction of secondary phases as well as the density and distribution of non-metallic inclusions.

The analysis of research and publications of the recent years shows that one of the primary structural elements affecting the structure of iron-carbon alloys are components of nanometric dimensions, the addition of which in small amounts makes it possible to significantly affect the structure and mechanical properties of the weld metal. A review of reference publications indicates the use of nano-additions as a way of shaping the structures of higher mechanical properties, which is important in the manufacture of critical elements made of high strength low-alloy steels.

The publications [1, 2] show that inclusion engineering can be used for optimising the microstructure of steel in order to improve its mechanical properties. Inclusions (oxides, sulphides and carbides) having the size <1 micrometre, favouring the formation of acicular ferrite (AF) have been treated as a separate group and named "dispersoids" as due to their small size they do not reveal any negative impact on mechanical properties, yet they determine the conditions for shaping the microstructure of metal.

The authors [3] have experimentally demonstrated the effect of non-metallic inclusion dimensions on the heterogeneous formation of acicular ferrite structure capable of ensuring weld metal mechanical properties of high plasticity level. The publication [4] contains the statement that the formation of titanium oxides and nitrides in weld metal having higher titanium and boron content favours the formation of AF.

Valeriy D. Kuznietsov, Professor Ph.D. (DSc) Eng. - Institute of Surface Engineering of Ukraine's National University of Technology, Kiev; Konstantin P. Szapovalov, "Novokramatorsky Mahynobudivny Zavod, Kramatorsk

The publications [5, 6, 7] contained information about experimentation and theoretical substantiation of using primarily nano-carbides for AF formation conditions and improving low-alloy steel weld metal mechanical properties. The conditions of structure formation as well as the composition and properties of non-metallic inclusion distribution in the presence of aluminium and titanium oxides were determined in the publications [8, 9], also stating the positive influence of such oxides on weld structure formation control. The same conclusions were formulated in the publication [10] while assessing the various volumetric contents of nano-oxides in the weld metal. Therefore, in order to shape the structure of welds having higher physico-mechanical characteristics it is necessary to ensure the formation of inclusions in metal, where such inclusions are forecast in terms of volume fraction, composition and dimensions.

The objective of this work was to investigate the effect of nano-oxides on the distribution of non-metallic inclusions and on the structure and properties of weld metal in welding high strength low alloy steels.

Tests and Results

The tests involved the use of the 10G2FB low-alloy high strength steel and the Sv-10HGN2SMFTJu filler metal wire. Nano-components were provided to the weld pool as mortar formed by pressing and sintering the homogeneous mixture of 40 μm fraction iron powders and aluminium or titanium nano-powders (60 nm) of pre-defined volumetric ratio. The mortar prepared was used as a filler metal in the form of an electrode having a specified length and diameter, placed in the weld groove prior to welding. Each of the mouldings was placed consecutively along the interfaces, which in single run welding excluded the effect of welding parameter changes on the result.

MAG welding was performed using an ADF 231 welding head and a KP 004 U3 semi-automatic welding machine and the following parameters: current $I = 170 \div 180$ A, arc voltage $U = 25 \div 27$ V, welding rate $V_s = 12.5$ m/h, shielding gas flow rate 9 l/min, wire extension 15 mm. The shielding gas used was the ISO 14175 – M31-ArC-28 (72% Ar + 28% CO_2) gas mixture. The chemical composition of the weld metal is presented in Table 1.

Table 1. Chemical composition of Sv-10HGN2SMFTJu filler metal wire weld metal

Component	C	Cr	Mn	Ni	Si	Mo	V	Ti	Al	P	S
Content,%	0.08	0.3	1.6	2.1	0.45	0.43	0.04	0.06	0.009	0.02	0.015

In order to assess the effect of inclusions on the weld structure formation, the welds were tested for their chemical composition as well as for the dimensions and density of inclusions. The inclusions were additionally identified using photographs in back-scattered electrons, due to which it was possible to identify the inclusions as inclusions and not as gas pores.

It was observed that the inclusions tended to have a spherical shape, yet it was also possible to notice irregularly-shaped inclusions (Fig. 1). The precipitation of inclusions was observed both in the grain and on the ferritic phase boundaries, which can indicate the possible role of an inclusion as a ferrite nucleus.

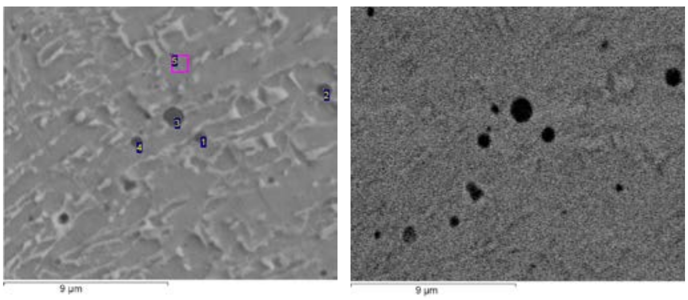

Fig. 1. Non-metallic inclusions in the weld metal after providing the weld pool with complex nano-oxide (56.5% Al_2O_3 – 41.5% ZrO_2 - 2%YO_2) – 0.5%: a – electron image x6000, image in back-scattered electrons

The results of the local spectral analysis of the chemical composition of inclusions revealed that, irrespective of dimension, in each of inclusions it was possible to observe a significant increase in carbon, oxygen, aluminium, sulphur, titanium and manganese contents (Table 2). The increase was particularly noticeable as regards carbon, oxygen and sulphur (by several orders of magnitude). The successive spectrum number in Table 2 corresponds to the separated spectrum of chemical elements in the inclusions marked with numbers on Figure 1.

Table 2. Contents of chemical elements in the inclusions according to Figure 1

Spectrum	C	O	Al	Si	S	Ti	Mn	Fe	Ni	In total
1	4.90	15.39	1.87	5.36	0.46	1.42	8.97	61.17	0.47	100.0
2	3.22	15.56	1.76	5.10	0.26	0.62	6.10	66.55	0.84	100.0
3	3.23	22.64	2.88	8.23	0.52	1.52	11.34	48.99	0.65	100.0
4	5.79	15.84	1.59	5.21	0.42	1.00	8.60	60.77	0.78	100.0
5	1.73	0.54	0.00	0.15	0.00	0.00	0.53	95.40	1.64	100.0
On average	3.77	13.99	1.62	4.81	0.33	0.91	7.11	66.58	0.87	100.0

In the case of the local spectral analysis of the inclusion being 0.4 μm in size, the tendency of chemical element content increase was maintained, yet it was lower in quantitative terms. This fact could result from the probe (1 μm in diameter) covering part of the solid solution matrix, where the content of chemical elements was significantly lower and close to their content in the weld metal, and not in the inclusion. The increased content, particularly of oxygen and aluminium confirmed the fact that the non-metallic inclusion nucleus was constituted by aluminium oxide.

The analysis of inclusion morphology indicated the complex structure (Fig. 2). On the inclusion periphery it was possible to observe other nano-build-ups arranged in the form of a blanket. On the basis of the analysis of the separated spectra it could be assumed that the inclusion peripheral zone included partly iron carbides as well as manganese sulphides and silicon sulphides.

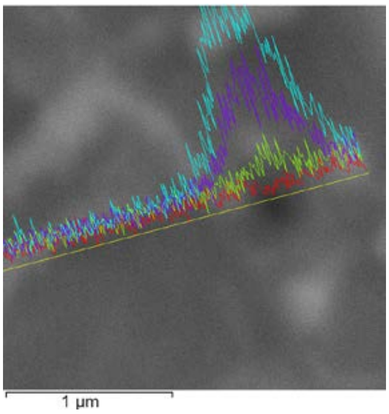

Fig. 2. Spectrum of chemical elements during inclusion scanning (red – carbon, yellow – silicon, violet – manganese, green – sulphur)

The computer-aided processing of the results related to the distribution of non-metallic inclusions according to dimensions enabled the separation of three basic dimensional groups: inclusions being up to 0.3 μm in size, from 0.3 to 0.8 μm and over 0.8 μm. On the basis of the analysis of the distribution of non-metallic inclusion amounts it was ascertained that in the initial state, without nano-additions, the volume fraction of inclusions amounted to 0.47%, in this the inclusions in the 0.3-0.5 μm range – 36% and in the 0.5-0.8 μm range – 31%. The basic mass of inclusions was constituted by the inclusions of up to 0.8 μm in size. In the weld metal with the TiO_2 – 0.5% nano-oxide, the volume fraction of inclusions amounted to 0.41%, in this the inclusions in the 0.3-0.5 μm range – 43% and in the 0.5-0.8 μm range – 26%. The basic mass of inclusions was also constituted by the inclusions of up to 0.8 μm in size. In the weld metal with the Al_2O_3 – 0.5% nano-oxide, the volume fraction of inclusions amounted to 0.44%, in this the inclusions in the 0.3 μm range – 29%, in the 0.3-0.8 μm range – 52%, and in the 0.5-0.8 μm range – 19%. The basic mass of inclusions was also constituted by the inclusions of up to 0.8 μm in size. The data analysis indicates an increase in the volume fraction of the inclusions from the 0.3-0.5 μm range and, accordingly, a decrease in the 0.5-0.8 μm range for the introduction of aluminium and titanium nano-oxides in comparison with the initial structure.

The determined correlations were also confirmed by the results of the analysis of only spherical inclusions according to the diameters of an equivalent circle. Figure 3 presents the bar charts according to the volume fraction and distribution of such inclusions in the weld metal for the initial state and with titanium nano-oxides.

The data processing according to the equivalent circle diameter indicator showed that in the initial state without the addition of oxide nano-powder the principal part of spherical inclusions from 4% to 6% coincides both with the size range up to 0.3 μm, as well as with the 0.3-0.5 μm range and beyond. It was also possible to observe inclusions to 2% of particles being up to 1.5 μm in size (Fig. 3a). After adding the oxide nano-powder of TiO_2–0.5% by volume part of the spherical inclusions being up to 0.3 μm in size as well as in the 0.3-0.5 μm range increased from 6% to 14%. The presence of the inclusions being over 0.8 μm in size was basically not detected (Fig. 3b).

The complex analysis of inclusions indicates the significant differences in their sizes and distribution density in the metal matrix with the presence of nano-oxides, thus the influence of the nano-oxides in the weld metal structure. In fact, the metallographic analysis results revealed that the most common ferrite morphological forms in the weld metal structure were block ferrite (BF), lamellar ferrite (LF), intragranular acicular ferrite (AF), Widmanstätten ferrite (WF), upper bainite (UB) and lower bainite (LB). Table 3 presents the percentage content of each of the forms in the welds tested.

The initial weld structure was characterised by the increased content of brittle components (block ferrite, Widmanstätten ferrite, upper bainite) and the formation of acicular ferrite having a high shape coefficient (L/B) amounting to 4÷7 and an acicula length up to 20 μm.

Table 3. Contents of characteristic ferrite morphological forms in the weld structure

Weld	Components of weld microstructure,%					
	BF	LF	AF	UB	LB	WF
Initial weld, without nano-oxides	up to 10	10-20	up to 10	20-40	20-40	up to 35
Weld with Al_2O_3 nano-oxide – 0.5% per volume	up to 20	up to 40		up to 20		up to 20
Weld with TiO_2 nano-oxide – 0.5% per volume	up to 10	up to 10	20-40	10-20	10-15	up to 15

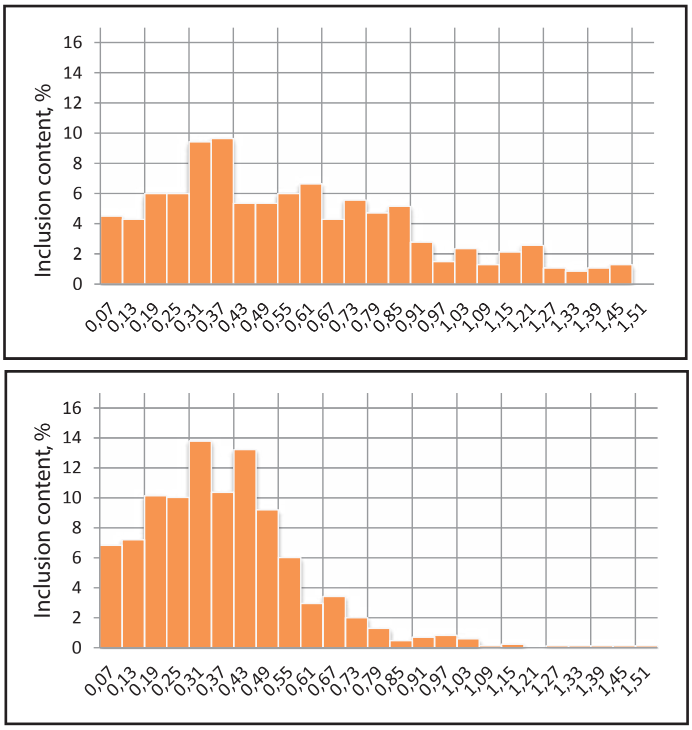

Fig. 3. Bar chart of inclusions distribution in the weld metal according to the equivalent circle diameter indicator, in μm: a) in the initial state, b) with the TiO_2 nano--oxide in the amount of 0.5% by volume.

The intragranular ferrite was formed both as the bainitic phase and as massive ferrite. On the grain boundaries ferrite precipitated in its acicular form and as Widmanstätten ferrite. The weld metal microstructure had the sufficiently high content of intragranular polygonal ferrite with allotriomorphic ferrite precipitates on grain boundaries (Fig. 4, a-c). The welds of such a structure are characterised by low level of ductility and metal plasticity.

The structure of weld metal with the Al_2O_3 nano-oxide – 0.5% by volume (Fig. 4, d-f), containing brittle components (block ferrite, Widmanstätten ferrite and upper bainite), was characterised by the formation of acicular ferrite having a high shape coefficient (L/B) amounting to 5÷10 and an acicula length up to 20 µm and by accordingly higher plasticity, which was demonstrated by structure component microhardness measurement results changing from 223 HV to 232 HV.

In comparison with the initial structure the structure of the weld metal with the TiO_2 nano-powder – 0.5% by volume was characterised by the lower content of brittle components (block ferrite, Widmanstätten ferrite and upper bainite) and by the formation of acicular ferrite having a more favourable shape coefficient (L/B) amounting to 3-5 and an acicula length up to 5 µm (Fig. 4, g-i). Welds of such a structure are characterised by sufficiently high level of metal ductility, plasticity and strength. The measurements revealed that the microhardness of structural components changed from 230 HV to 250 HV. The tests conducted have revealed that providing the weld pool with nano-oxides, particularly titanium, leads to positive structural changes as regards the ductile morphological forms of ferrite due to the significant presence of inclusions being up to 0.3 µm in size.

Table 4. Results of the mechanical tests of joints made of the A514 steel using the Sv-08G2S wire

Welding variant	Yield point Re, MPa	Tensile strength Rm, MPa	Relative elongation,%	Relative area percentage reduction,%	Toughness KCV, J/cm²
Without nano-additions	357	542	21	61	46
With TiO_2 nano-oxide – 0.5% per volume	514	647	12	54	93
With Al_2O_3 nano-oxide – 0.5% per volume	535	668	17	60	48

The results of laboratory tests were confirmed during production tests performed at Novokramatorsky Mashinobudivny Zavod using the MAG method for welding low-alloy high strength A514 steel (equivalent S690Q). The tests also aimed to verify the possibility of using cheaper welding consumables related to providing the weld pool with nano-oxides. To this end, the A514 steel was welded with the most commonly used and the cheapest filler metal wire Sv-08G2S (ISO 14341-A-G3Si1) having a diameter of 1.2 mm. The results of mechanical tests are presented in Table 4.

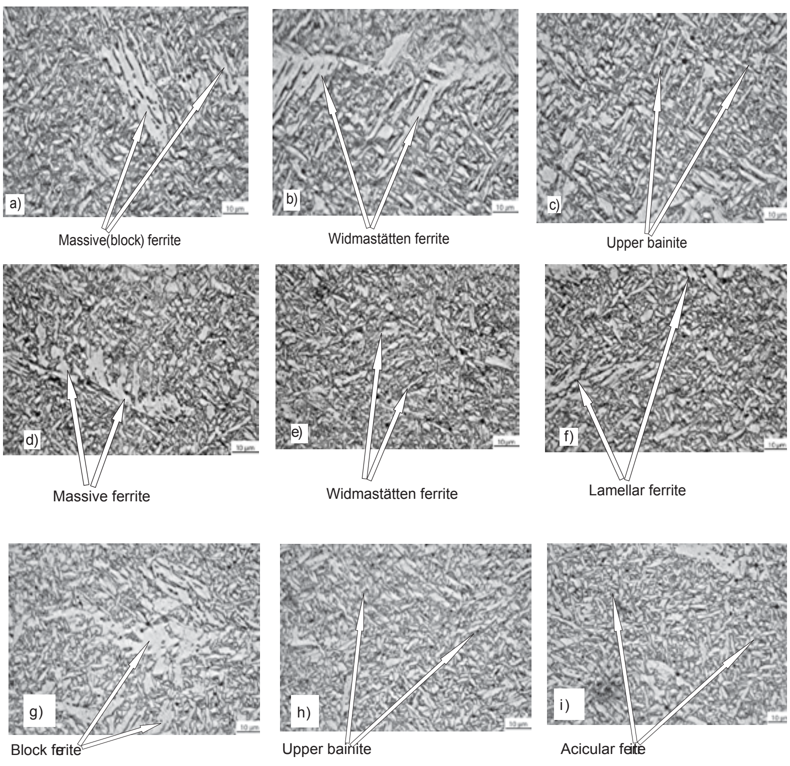

Fig. 4. Characteristic structures of weld metal without nano-oxides (a, b, c); with the Al_2O_3 nano-oxide – 0.5% by volume (d, e, f); with the TiO_2 nano-oxide – 0.5% by volume (g, h, i). The arrows indicate characteristic ferrite morphological forms

Table 4 indicates an increase in the yield point and that in the tensile strength when the weld pool was provided with titanium and aluminium nano-oxides. The indicators above were most significantly affected by the aluminium nano-oxides, increasing the yield point by 178 MPa and the tensile strength by 126 MPa. Comparing these data with the test results concerning the joint welded with the more expensive Sv-10HN2GSMFTJu grade wire having a tensile strength of 710 MPa it is possible to observe that while using the

Sv-08G2S wire for welding the A514 steel this indicator was close to $R_m = 668$ MPa. While providing the weld pool with the titanium nano-oxide it was possible to observe that its influence on the yield point and tensile strength was lower, yet the toughness value increased two times if compared with that observed in the initial material.

Concluding remarks

1. It was determined that the initial weld metal structure contained non-metallic inclusions mainly in the 0.3-0.5 µm size range and beyond, whereas in the case of nano-oxides introduction the amount of the inclusions increased both in the 0.3-0.5 µm size range and in the size range up to 0.3 µm. The inclusion nucleus was constituted by the aluminium or titanium oxides. The peripheral zones also included iron carbides as well as manganese sulphides and silicon sulphides.
2. It has been demonstrated that providing the weld pool with nano-oxides while welding low-alloy steels favours the formation of the plastic morphological varieties of, particularly acicular, ferrite in the weld metal structure due to the significant amount of non-metallic inclusions being up to 0.3 µm in size.
3. The production test results have revealed that nano-oxides in the amount of 0.5% by volume improve the mechanical properties of low-alloy steel weld metal, where the increase accompanying the use of the cheaper Sv-08G2S (G3Si1) filler metal wire is close to the values ensured by more expensive wires, e.g. Sv-10HN2GSMFTJu.

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