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Weld Metal Dendritic Structure Modification by Dispersed Refractory Oxide Particles

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Abstract

The paper deals with the modification effect of dispersed oxides particles (Al_2O_3, MgO, ZrO) on the dendrite structure in low-alloy weld metal. The flux-core wire 1.6 mm diameter for inoculating of oxides powder to weld pool was purposed. Obtained results confirmed an influence of inoculated refractory oxides on dendrites size and morphology, microstructure and mechanical properties of the weld metal.

Subject Areas

Metal Material

Keywords

Arc Welding, Low-Alloy Steel, Weld Metal, Modification by Refractory Oxides, Dendrite Structure, Mechanical Properties

1. Introduction

Currently, welding remains one of the most widespread technologies in the manufacture of metal structures. A wide variety of metals, types of welded joints and technologies used for these purposes led to the development of computer modelling with the "composition-structure-properties" system. This approach makes it possible to effectively and reliably select such a combination of technological parameters of the welding process, which will provide the required level of mechanical properties of the welded joint. At present, programs have become widespread, which are based on modelling the composition of the secondary structure of the weld metal [1] [2] [3]. In this case, the conditions for the formation of the structure as a result of recrystallization are considered, while the parameters of the primary structure are determined by an experimental or computational-experimental methods. This approach does not allow to take into account the influence of the initial process parameters on the nucleation and growth of primary grains, which leads to some uncertainty in modelling the final results. The formation of the structure of the weld is a continuous fast-flowing process that begins with the melting of the base and filler materials and ends with the cooling of the metal to ambient temperature. Based on this approach, the modelling of the structure should begin with consideration of the conditions for the nucleation and growth of dendrites in the weld pool, the formation of grains of the primary structure, and end with the formation of the secondary structure. Unfortunately, there are not many investigations of the initial part of this process [4] [5] [6].

The weld metal in arc welding of carbon and low-alloy steels in accordance with the equilibrium phase diagram (Fe-C diagram) demonstrates the partial solubility of alloying and impurity elements in solids with a peritectic reaction. The microstructure of the solidification of the weld pool begins with the formation of equiaxed or columnar dendrites, regardless of the welding conditions and deoxidation process.

Dendritic growth is a fundamental crystal growth phenomenon that accompanies most casting and solidification processes. Dendrites are a ubiquitous crystalline form in solidifying alloys and supercooled melts, as their shape is most suitable for efficient heat and mass transfer on a small scale. The formation of morphological forms of dendrites is associated with the development of such chemical processes as microsegregation, thermal (release of latent heat), and mechanical processes (changes in volume during phase transformation). All these interactions take place at the interface between dendrites and melt. Therefore, understanding the growth of dendrites is considered important for the management of the basic processes of crystal formation and growth.

The dendritic structure is characterized by two parameters: the distance between the axes of the dendrites and the segregation coefficient, which represents the ratio of the highest and lowest concentrations of an element in the axes and in the inter-axial spaces of the dendrites. The value of the coefficient of dendritic segregation is influenced by the speed of the crystallization front and the temperature gradient at this front. With a decrease in the thickness of the dendrite, the efficiency of diffusion redistribution of alloying and impurity elements increases. The effect of the size of dendrites on the heterogeneity of the metal composition is especially noticeable at presence in the melt of such strongly liquidating elements as molybdenum, chromium, manganese, sulfur, phosphorus. These elements cause a change in the temperature range of crystallization, the critical cooling rate of austenite, and the formation of structural inhomogeneity. Structural heterogeneity, in turn, leads to anisotropy of plasticity and toughness of the weld metal.

Surface energy and surface tension are considered fundamental quantities for understanding surface phenomena at the boundaries of growing dendrites. The concepts of surface free energy (E) and surface tension (σ), introduced by Gibbs, are the two main macroscopic parameters characterizing the thermodynamic properties of crystal surfaces of dendrites.

Although there was pointed out a century ago that the increasing thermal excitation of atoms in solids leads to decreasing in the work of creating the surface (surface energy), the temperature dependences of σ are still largely unknown due to the lack of direct measurements. In most cases, the available absolute values of the surface energy were obtained at temperatures slightly below the melting point (T_m), where the plastic flow of the metal occurs, which is determined by the surface tension of the liquid phase.

A decrease in dendritic segregation and the associated anisotropy of metal viscosity should begin with a change in the composition of the melt before crystallization. The introduction of small particles of solid impurities into the weld pool whichsizes correspond to the parameters of incipient dendrites (nanomodification), creates new crystallization centers and promotes dendrite grinding.

Features of the dendritic structure contain important information about the processes of crystallization and structure formation in real volumes of metal and largely determine the level of physical and mechanical properties of welded joints. That nanomodification during crystallization makes it possible to purposefully affect on the dispersity and morphology of the cast structure and the phase-structural state of the weld metal. The features of the dendritic structure incorporated during crystallization retain their influence on the structure and properties after the completion of the recrystallization process and the formation of the secondary structure. Based on this, understanding the features of the formation of a dendritic structure under conditions of crystallization of the weld pool is one of the main criteria for assessing and predicting the properties of the weld metal.

Formation of weld metal structure starts from the dendrite phase nucleation and growth processes in a melted weld pool metal. Growing dendrites accumulate the refractory inclusions on the boundaries "fused-solid" in the melt. Size reduction of inclusions leads to rise of relationship between the number of particles on their surface and in volume, respectively, to increase in the energy of particle interaction with melt that promotes enhancement of their efficiency as modifiers.

Application of dispersed inclusions is perspective from this point of view [7] [8]. It was shown that addition into a steel melt of 3 - 30 μm size refractory particles results in formation on their surface of cluster shells of up to 30 - 60 μm thickness [9] [10] [11]. It is proved by decrease of melt toughness index. Such clusters can be effective centers of new phase nucleation in the melt and promote change of dendrite morphology [12] [13] [14] [15]. The aim of the present work was to study the possibility to effect on dendrite structure due to the dispersed non-metallic inclusions for modification of low-alloy high-strength steels weld metal microstructure. This study is aimed at expanding our knowledge base on the features of the formation of dendrites in the weld metal of low-alloy steels.

2. Materials and Experimental Procedure

The welds were prepared by the flux-core arc welding technique. The joint geometry is illustrated in **Figure 1**. There were used the wires of 1.6 mm diameter and shielding gas M21 according to ISO 14175-2010. Welding was carried out using 240 - 250 A reverse polarity direct current at arc voltage 31 - 32 V and welding rate 10 - 12 m/h. The flux combinations of wires were constant for produce welds (designated 0, Al_2O_3, MgO, ZrO_2) with approximately identical carbon, manganese and silicon concentrations, but with different inclusions concentrations in the microstructure. **Figure 2** shows a scheme of a groove filling, according to which passes 1 - 8 were carried out in welding using a wire of basic alloying system, and in performance of passes 9 - 16 a flux-cored wire of 1.6 mm diameter having a core with particles of dispersed refractory compounds was introduced in a weld pool in form of cold filler. The particles of aluminium, magnesium and zirconium oxides of 20 - 60 μm size were used as modifying additives as shown in **Figure 3**. The basic alloying system C-Mn-Cr-Ni-Mo-Si-Cu provided formation of weld metal with ferrite-bainite structure, which on their mechanical properties corresponds to low-alloy steels of K65 strength category. The transverse samples for investigation of structure and phase composition of weld metal as well as mechanical properties of welded joints on scheme presented in **Figure 4** were cut out of the welded joints.

Metallographic investigations were used to determine the weld metal composition, the fraction of separate constituents of its microstructure, the volume fraction and the size distribution of non-metallic inclusions. Examination of structure and non-metallic inclusions were carried out by methods of optical and electron microscopy using Neophot-30 microscope, electron scanning microscope JSM 35CF with attachment for local X-ray analysis INCA Energy 350 as well as computer programs developed at the E.O. Paton Electric Welding Institute for analysis of microstructure constituents and distribution of non-metallic inclusions by size and composition.

Quantitative determination of microstructural constituents was carried out in accordance with ASTM El 12 - 12 procedure. Microhardness of separate structural constituents was measured on M-400 hardness device of LECO Company at 100 g loading and integral hardness was determined on Vickers at 1 kg loading. A digital image was obtained using Olympus camera.

The weld metal primary structure was examined on polished samples etched in boiling saturated solution of sodium picrate in water. A microstructure of the last pass of metal in the multipass weld (*i.e.* cast structure) was examined. The samples were cut out in a direction normal to weld longitudinal axis in such way that on the surface of the section it was possible to see the dendrites, which grew in direction of the highest thermal gradient in a weld pool. Sizes of columnar dendrites (X, sizes shown in **Figure 5**) were determined in investigation of the primary structure on images obtained by method of optical microscopy.

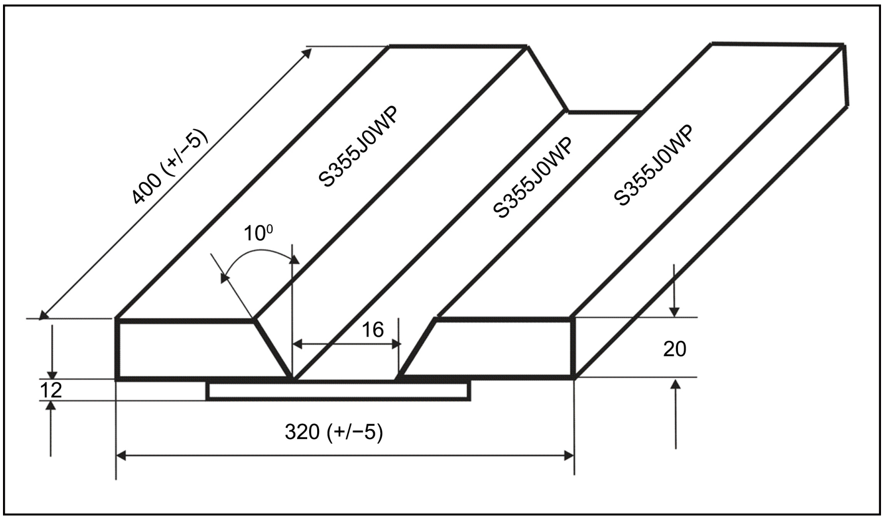


Figure 1. Scheme of the welds preparation.

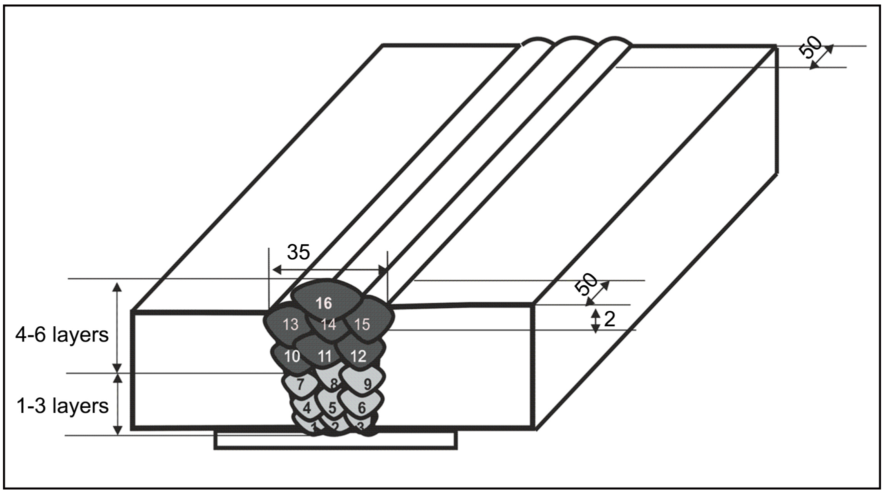


Figure 2. Scheme of the butt joint filling.

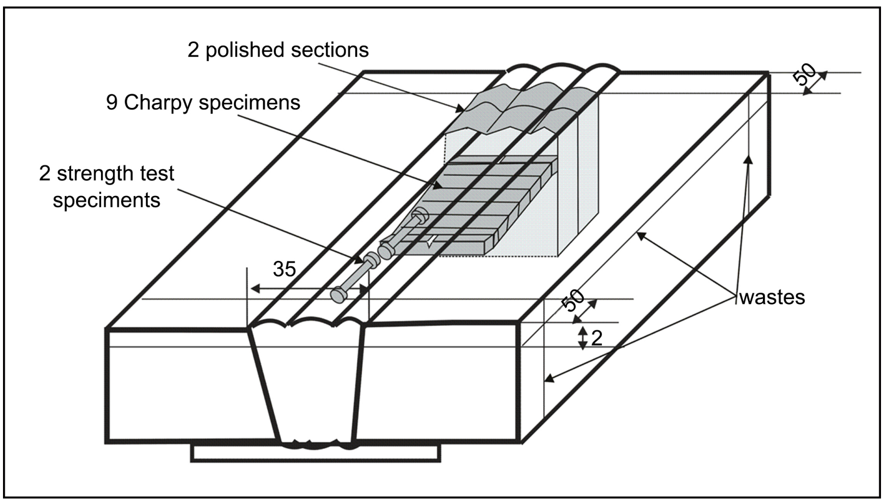


Figure 3. Scheme of samples cutting for investigation of chemical composition, mechanical properties and microstructure of the weld metal.

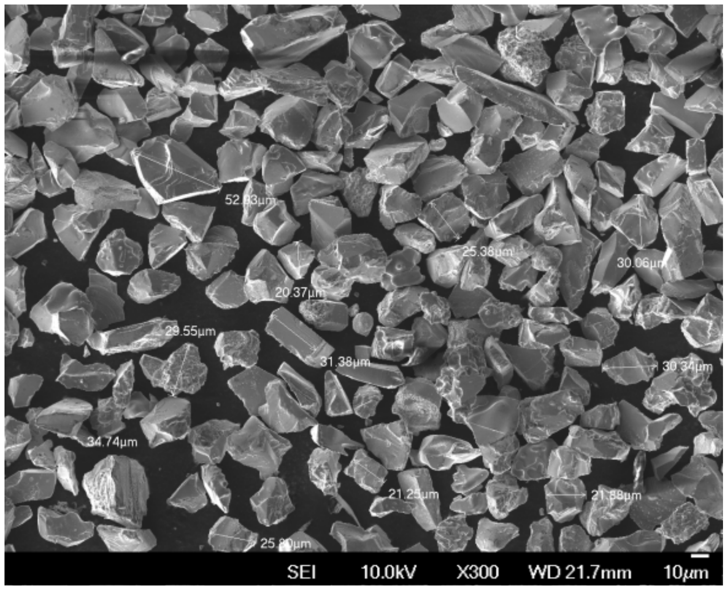


Figure 4. Particles of refractory oxides introduced into the weld pool.

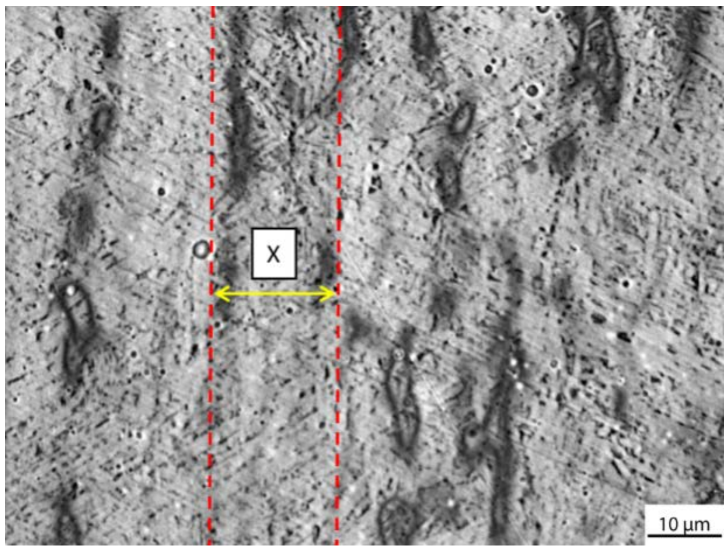


Figure 5. Scheme of determining the distance between the dendrites axes.

3. Results and Discussion

Results of composition and mechanical properties of the examined welds are presented in Table 1 and Table 2. Metallographic analysis revealed that microstructure of the examined welds contains products of austenite decay in process of metal cooling and some amount of non-metallic inclusions. Figure 6 provides the histograms inclusions distribution, which were obtained as a result of inclusions sizes analysis in the examined welds. Total fractions of non-metallic inclusions (F_{in}) are shown in Table 3. Metallographic analysis of the weld metal microstructure with using optical and electron metallography methods showed that each grain of the primary structure contained two or more structural constituents of secondary structure.

Table 1. The chemical composition of the weld metal.

Modifier	C	Si	Mn	S	P	Cr	Ni	Mo	Al	Ti	Zr
0	0.042	0.340	1.19	0.021	0.020	0.11	2.13	0.28	0.028	0.029	-
Al_2O_3	0.034	0.424	1.40	0.017	0.023	0.12	2.15	0.29	0.032	0.015	-
MgO	0.031	0.227	1.11	0.025	0.024	0.14	1.85	0.29	0.023	0.030	-
ZrO_2	0.033	0.223	1.05	0.024	0.024	0.12	2.02	0.30	0.024	0.031	0.06

Table 2. Mechanical properties of the weld metal.

Modifier	σ_B	$\sigma_{0,2}$	δ	ψ	KCV, J/cm^2 at T, ˚C				
	MPa		%		+20	0	−20	−40	−60
0	693	605	14	49	97	87	75	53	37
Al_2O_3	728	621	17	54	82	58	50	36	22
MgO	644	586	19	60	103	85	69	60	34
ZrO_2	622	533	19	65	120	107	73	65	41

Where: σ_B—tensile strength; $\sigma_{0,2}$—yield strength; δ—% of elongation; ψ—% of reduction area; KCV—impact properties.

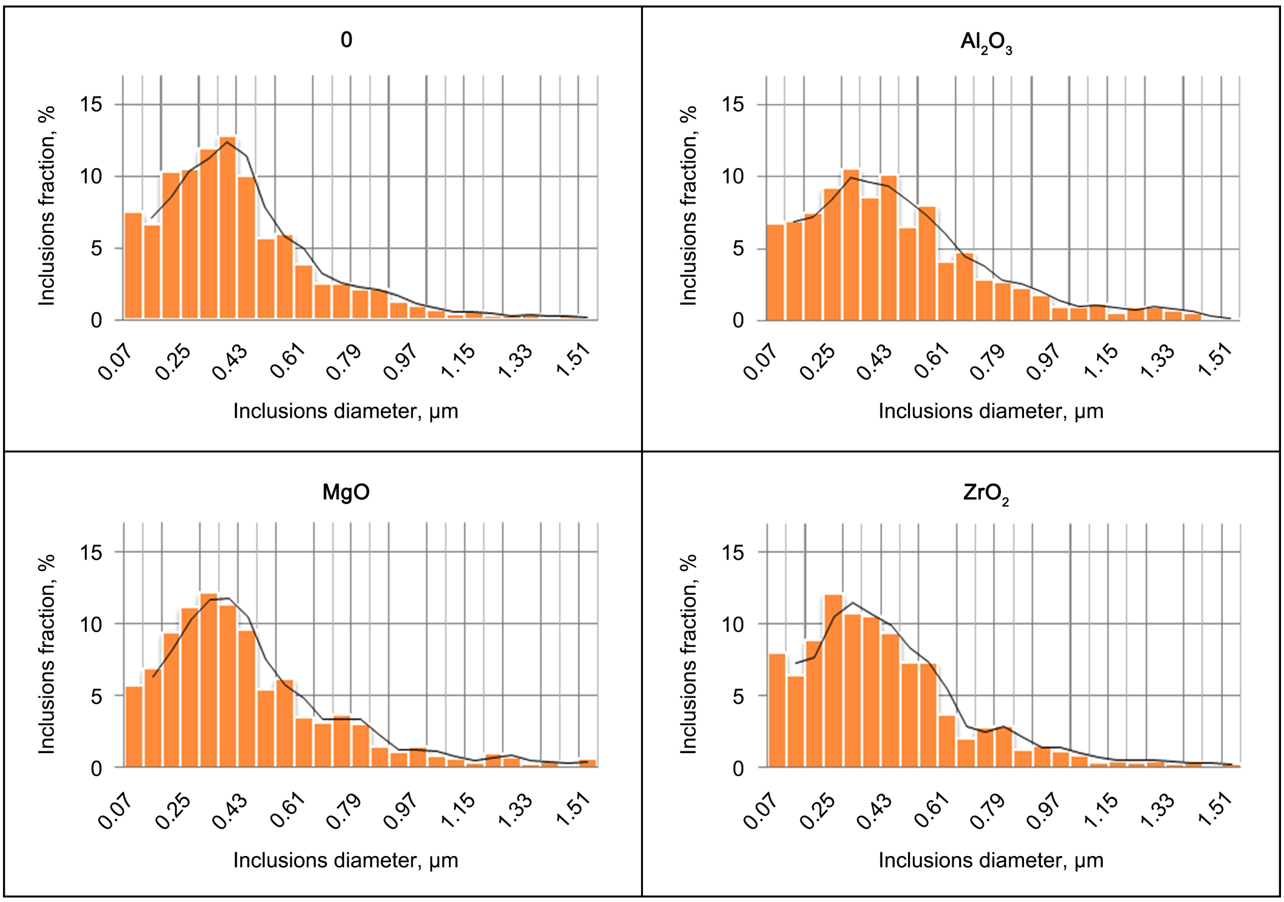


Figure 6. Histograms of the non-metallic inclusions size distribution in the weld metal.

The most common secondary structures observed in the weld metal were grain-boundary allotriomorphic ferrite (GBF), intragranular polygonal ferrite (IPF), globular ferrite (GF), Widmanstatten ferrite (WF), acicular ferrite (AF), upper and lower bainite (UB and LB), phase containing martensite, austenite and carbides (MAC). The content of the main structural components of the weld metals are given in Table 4.

The results of distance measurement between the dendrite axes in the investigated welds metal structure are shown in Table 5. They demonstrate significant differences in the dendrites morphology depending on the modifying additive as shown in Figure 7.

The structure of weld metal, which did not inoculate modifiers in the weld pool (weld metal index—0), is characterized with high content of non-metallic inclusions with size not more than 0.3 μm as shown in Figure 8. The grain boundaries are well-pronounced and have elongated morphology. Ferrite precipitates along the grain boundaries mostly in the form of Widmanstatten ferrite. Microstructure observed in the grain body consisted mainly of intragranular ferrite and lower bainite precipitations as shown in Figure 9. Such structural composition provides high levels of weld metal strength (at the level of steels of K70 strength category) and sufficiently high level of viscosity and impact toughness as shown in Table 2.

Table 3. Volume fraction of non-metallic inclusions in the weld metal.

Modifier	0	Al_2O_3	MgO	ZrO_2
V_{incl}, %	0.42	0.74	0.62	0.55

Table 4. The content of the main structural components in the weld metal.

Modifier	AF	GBF	IPF	GF	WF	UB	LB	MAC
0	8	5	8	2	15	40	17	5
Al_2O_3	2	2	8	4	30	36	11	7
MgO	32	10	5	10	7	12	19	5
ZrO_2	30	15	2	6	7	10	25	5

Table 5. The results of measuring the distance between the dendrites axes.

Modifier	Distance between the dendrites axes, μm	Average value
0	50; 50; 60; 25; 40; 50; 45; 50; 40; 55	46
Al_2O_3	50; 30; 30; 40; 45; 30; 50; 40; 30; 30	57
MgO	140; 150; 120; 140; 90; 120; 100; 130; 80; 150; 300	152
ZrO_2	240; 200; 150; 140; 120; 120; 200; 80; 240; 90	158

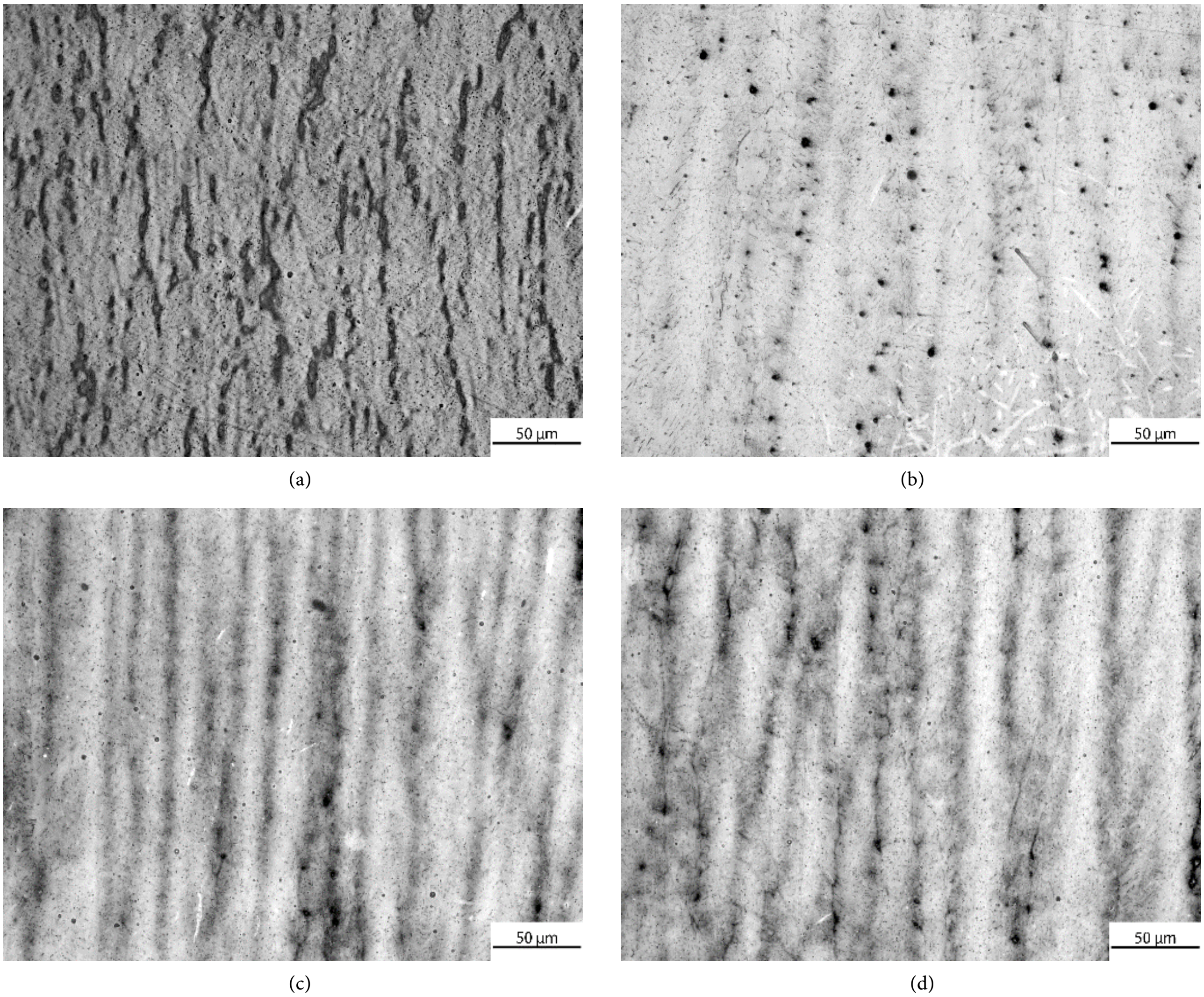


Figure 7. Dendritic structure of the weld metal (X320). (a) without modifier; (b) Al_2O_3; (c) ZrO_2; (d) MgO.

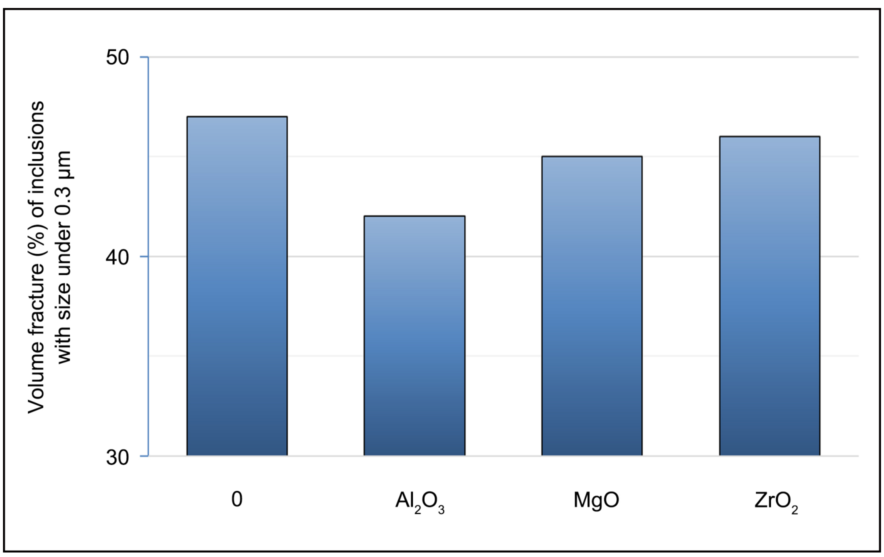


Figure 8. The fracture of non-metallic inclusions with size < 0.3 μm in the weld metal.

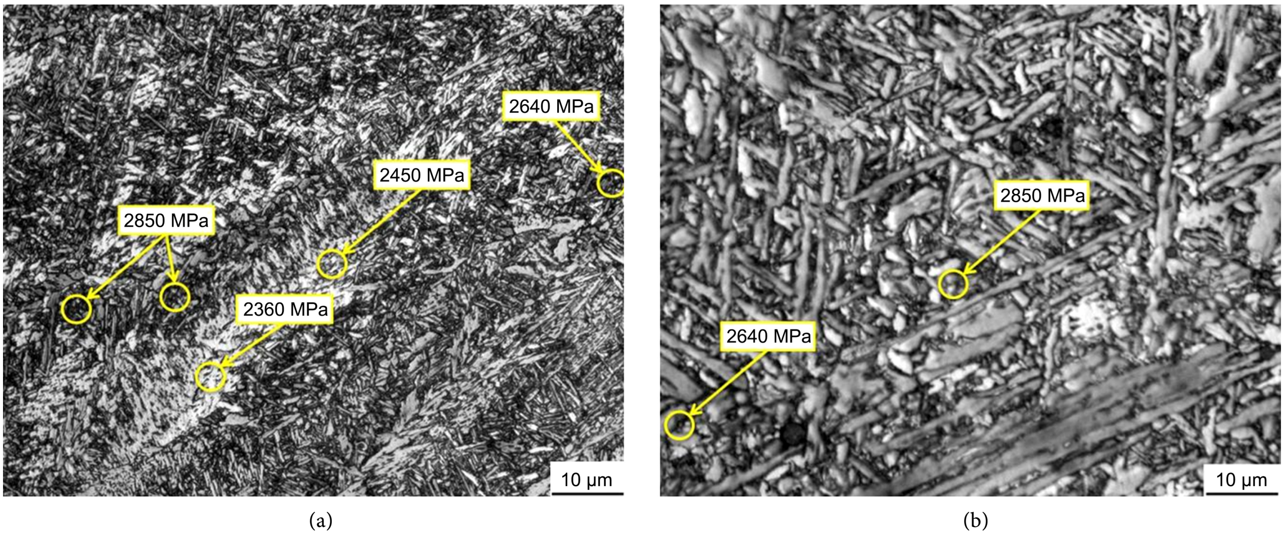


Figure 9. Microstructure and microhardness (HV1) of the structural components in the weld metal without modifiers: (a) X320; (b) X1000.

Sufficiently high fraction of nonmetallic inclusions of up to 0.3 μm size as shown in Figure 8 is kept in weld metal with magnesium oxide inoculating into the weld pool (weld metal index—MgO). The content of magnesium oxides was not revealed in the composition of nonmetallic inclusions. A weld metal microstructure is characterized with high content of intragranular polygonal ferrite with small content of acicular ferrite. Ferrite on the grain boundaries precipitates in form of small fringes of allotriomorphic ferrite with reduced level of microhardness and Widmanstatten ferrite as shown in Figure 10. Such composition of structural elements results in significant increasing of the weld metal ductility in comparison with basic—alloying system and insignificant drop of impact toughness as shown in Table 2.

Modification of the weld metal with aluminum oxide leads to decrease of non-metallic inclusions content with size less than 0.3 μm as shown in Figure 8. Aluminum compounds were detected in inclusions of the entire size range. The weld metal microstructure is characterized with high content of intragranular polygonal ferrite and lower bainite with frequent inclusions of upper bainite. Also there is a Widmanstatten ferrite content increasing on grain boundaries as shown in Figure 11. Such structural composition is characterized with increased level of the weld metal strength as shown in Table 2.

In the weld metal with modification of zirconium oxide particles there is an increase of non-metallic inclusions fraction with not more than 0.3 μm size as shown in Figure 8. The content of zirconium oxides was not detected in the composition of nonmetallic inclusions. The weld metal microstructure is characterized with intragranular polygonal ferrite high content in combination with presence of upper and lower bainite. Massive precipitations of ferrite are observed on the grain boundaries as shown in Figure 12. This structural composition provides the mechanical properties of the weld metal by a combination of high indices ductility and impact toughness as shown in Table 2.

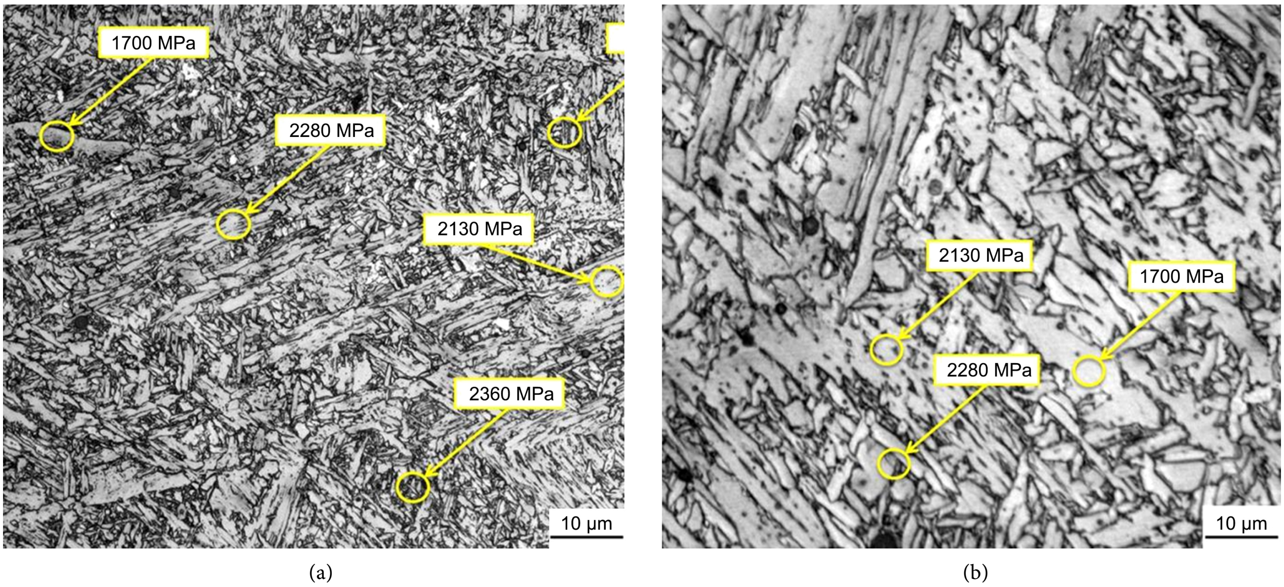


Figure 10. Microstructure and microhardness (HV1) of the structural components in the weld metal modified by MgO particles: (a) X320; (b) X1000.

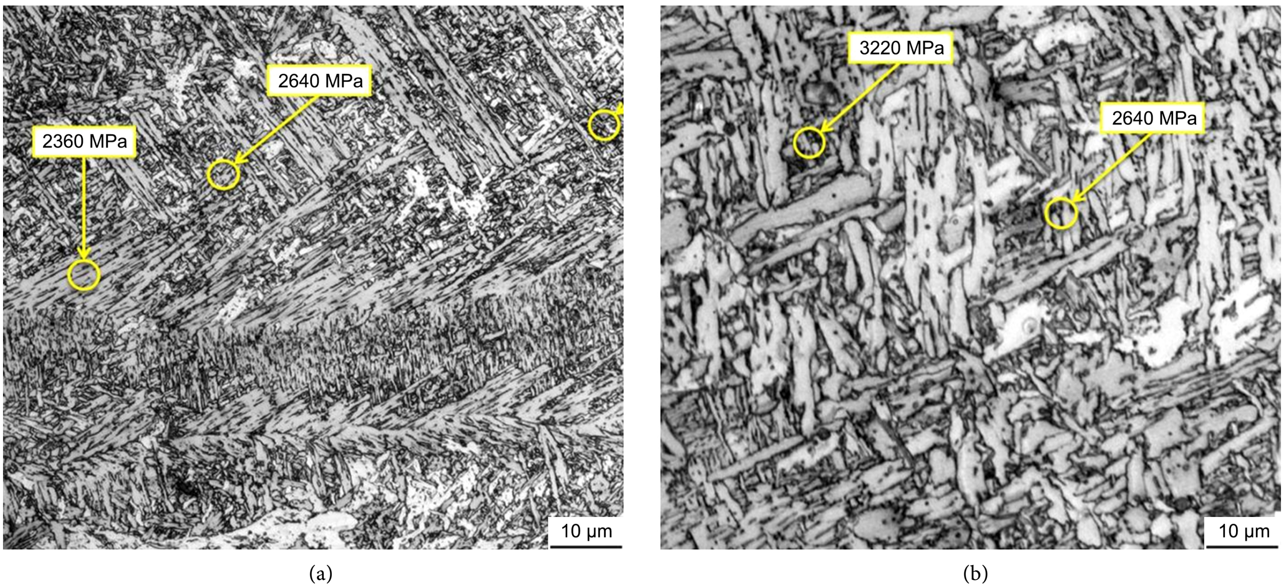
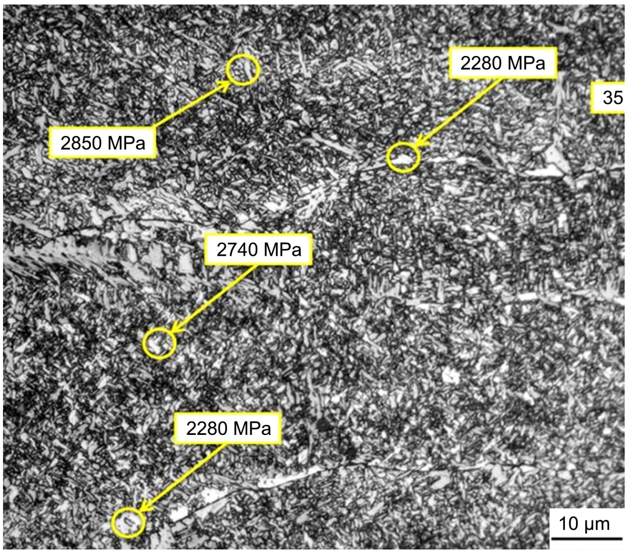


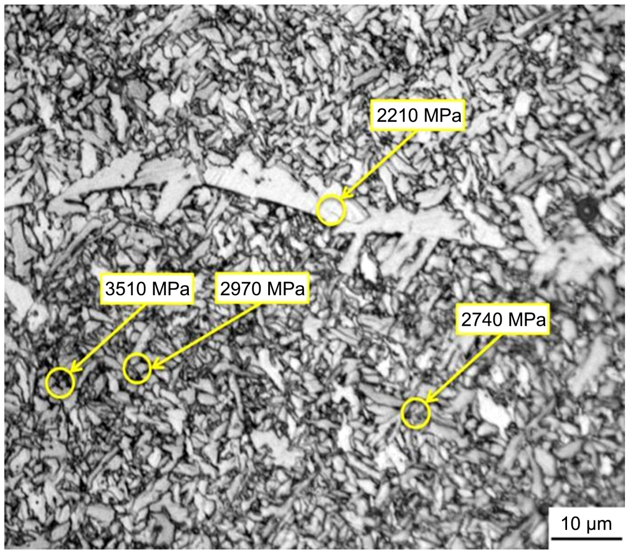
Figure 11. Microstructure and microhardness (HV1) of the structural components in the weld metal modified by Al_2O_3 particles: (a) X320; (b) X1000.

4. Analysis of Obtained Data

The analysis of experimental results was based on general ideas of dendrites nucleation and growth mechanism in the metallic melts. Today, there is considerably large amount of models describing these processes that indicates absence of some single approach, which would allow considering all complex of difficult and interrelated phenomena in the melts solidification process. It is generally accepted that there should be specific solidification centers in the melt in order to start this process. The debates hold on the issue what should be considered as



(a)



(b)

Figure 12. Microstructure and microhardness (HV1) of structural components in the weld metal modified by ZrO_2 particles: (a) X320; (b) X1000.

such centers. Two approaches to solution of this problem are widely presented in scientific literature. In accordance with one of them such centers can be refractory non-metallic inclusions [A], from other point of view, cluster formations can initiate solidification [BC]. Following from the considerations of thermodynamics the process of crystals nucleation in the metallic melt is possible under two main conditions: firstly, solidification centers should be with size more than critical size of nucleus, secondly, interphase energy on nucleus boundary with melt should be minimum [DF].

Globular nucleuses with minimum interphase energy can be the clusters of metal, presence of which in the melt was shown on practice [16]. Small size of such clusters (approximately 2 - 10^{-9} m) causes their high surface activity. The clusters can sorbing structurally free atoms of melt and promotes formation of micelles that was proved by experiments in investigation of refractory oxides effect on of liquid metals toughness [17].

Melting pool in arc methods of welding contains large amount of refractory inclusions, which size significantly exceeds a nucleus critical radius for iron melts solidification (approximately 4×10^{-7} m). Such inclusions are characterized with sufficiently high interphase energy on the boundary with metallic metal and, as a rule, do not satisfy the principle of structure-size correspondence in relation to iron crystals. Fused boundaries of base metal grains can be much more effective nucleuses for formation of new phase, however, following the requirements of the minimum interphase surface energy, 2D nucleuses thermodynamically lose 3D ones.

Table 6 shows the results of dendrite size determination in comparison with data on thickness of adsorption cluster shells forming on the surface of inclusions at 1600˚C temperature obtained in work [5].

Table 6. Comparison of measuring the of dendrites width with the interfacial tension (energy) at the metal-inclusion interface.

Modifier	Clustershell thickness, μm	Interfacial energy at the metal-inclusion interface, mJ/m^2	Contact angle at the metal-inclusion interface, deg	Dendrite width, μm
0	29	-	-	46
Al_2O_3	43	630	130	57
MgO	51	502	108	152
ZrO_2	59	470	106	158

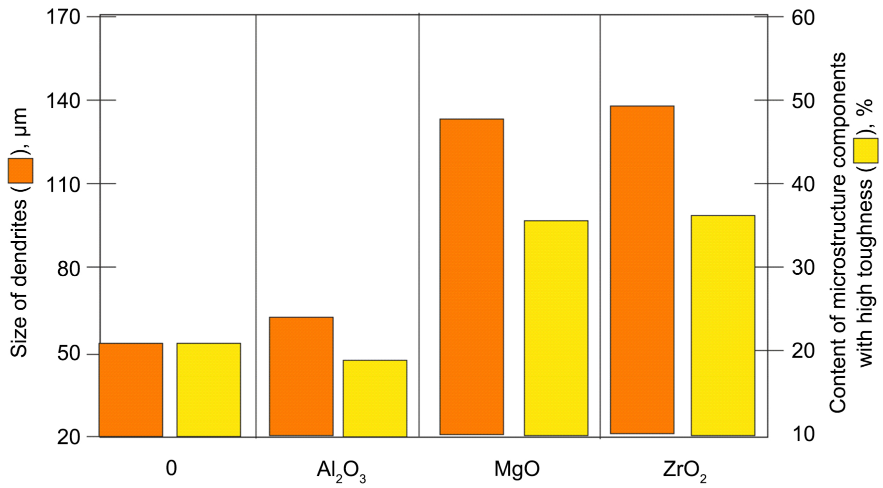


Figure 13. Connection between the size of dendrites and the content of microstructure components in the secondary structure of the weld metal.

As can be seen from given data there is a specific dependence between the morphology of dendrites and physical-chemical peculiarities of interphase boundary structure in the system "metallic melt-oxide inclusion".

In contrast to the classical theory of modification, which is based on the processes of heterogeneous nucleation of new phases with the participation of stable non-metallic inclusions, the authors of a number of works [6] [7] [8] [18] [19] believe that inclusions can have a modifying effect on metal melt clusters. The stability of the existence of clusters in a metal melt at crystallization temperatures for a time sufficient for a modifying effect on the formation of dendrites has been confirmed experimentally [7]. The interaction of positively charged clusters with oxygen anions in liquid metals promotes positive cluster adsorption [8]. In the weld metal modified with Al_2O_3 particles, aluminum compounds were found both in the form of individual inclusions with a size of less than 0.3 μm, and as part of larger inclusions of a more complex composition. An analysis of the composition of nonmetallic inclusions in the weld metal modified with MgO and ZrO_2 particles showed the absence of magnesium and zirconium compounds in them. The process of dissolution of these oxides should be accompanied by an increase in the content of large oxygen anions, which contributes to the positive adsorption of clusters and, accordingly, the formation of whiter large dendrites, which is confirmed by the data given in **Table 5**.

Increase of dendrite width promotes corresponding changes under conditions of secondary structure formation, content presented by increase of fraction of low-temperature constituents of bainite in weld metal structure (as shown in **Figure 13**) and changes level of its strength and toughness as shown in **Table 2**.

5. Conclusions

The paper was investigated the effect of refractory dispersed non-metallic inclusions inoculation into the weld pool on weld metal dendritic structure formation. The results obtained allowed us to draw the following conclusions.

The presence of a certain relationship between the dendrites morphology and the structure physicochemical features on the interface in the system "melt-oxide inclusion" was shown.

It was noted that the inclusions inoculation with a lower interphase tension and contact angle into the weld pool has a modifying effect on the dendritic structure in the weld metal.

An increase in the width of dendrites causes corresponding changes in the conditions of secondary structure formation, which is manifested in an increase in the low-temperature components of the bainite transformation in the weld metal structure and a change in their strength and toughness.

The results confirmed the possibility modifying the dendritic structure of the weld metal with using dispersed refractory particles compounds and controlling the secondary structure composition and the mechanical properties of welds metal. For a more full understanding of the dendritic structure formation processes, further development of work in this direction is required.

Conflicts of Interest

The authors declare no conflicts of interest.

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