About the problems of low technological plasticity of steel 04H14T3R1F used in the production of pipes for nuclear energy

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Abstract. *The features of the chemical and phase composition, structure and transformations in boride inclusions in the steel 04H14T3R1F, used for the manufacture of hexagonal pipe covers used during transportation to the place of regeneration and storage of spent fuel assemblies (FA) of nuclear power plants, have been studied. It has been established that steel 04H14T3R1F contains two types of boride inclusions $(Ti,Fe,Cr,V)_2B$ with a shell of $(Ti,Cr,V)_2B$ and $(Fe,Cr)_2B$, which have significant chemical heterogeneity. It is shown that in the process of hot deformation the phase and structural transformations occur: a change in the composition of borides due to the redistribution of elements, dynamic diffusion fragmentation and release of "satellite" particles, brittle destruction of borides, boride transformation. The behavior of boride inclusions and their influence on the mechanical properties of 04H14T3R1F steel at different plastic deformation temperatures was studied.*

KEYWORDS:: *SPENT FUEL ASSEMBLIES (FA) OF NUCLEAR POWER PLANTS, STEEL, BORIDE INCLUSIONS, DEFORMATION, PHASES, DIFFUSION, "SATELLITE" PARTICLES, DESTRUCTION.*

1.Introduction.

For transportation and storage of spent nuclear fuel, containers made of 04H14T3R1F steel alloyed with boron are used [1,2]. When boron is introduced into steel, a large quantity of borides is formed, which negatively affects its mechanical properties and technological ductility during the production of hot-rolled pipes [3-5]. The **goal of the work** was to study the influence of boride inclusions on the mechanical and technological properties of 04H14T3R1F steel.

2. Materials and Procedures.

The studies were carried out on the samples taken from pipe blanks, as well as from hot-rolled pipes made of steel 04H14T3R1F (Table 1). Deformation by oblique rolling was carried out in the temperature range 850...1150 ℃ (feed angle 5º30', roll rotation speed – 40 rpm). The influence of rolling parameters on the development of fracture processes under dynamic loading was assessed by the value of impact strength according to GOST 9454-98. Tensile tests were carried out according to GOST 1497, in addition, tensile tests were carried out at elevated temperatures (Instron 1195).

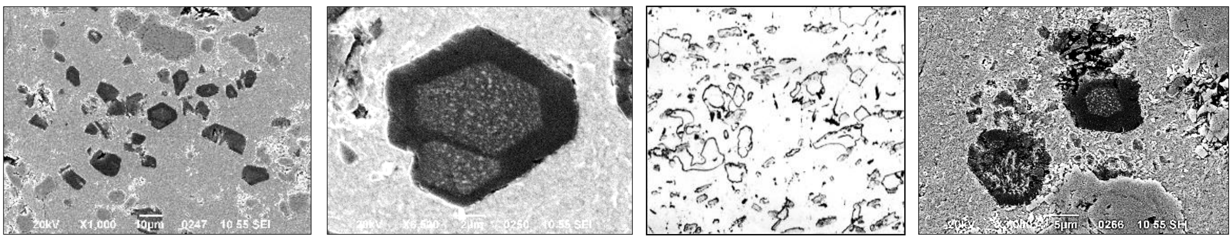
The microstructure of steel was studied using an optical microscope "Neophot - 31" and a scanning electron microscope JSM 35. The phase composition of steel 04H14T3R1F was determined by the X-ray diffraction method (DRON-2.0). Identification of boride inclusions was carried out using the metallographic method [6], as well as using a REMMA 102-02 microanalyzer. Thermogravmetric analysis was carried out and DTA curves were recorded.

Table 1. *Chemical composition of steel 04H14T3R1F*

Content of chemical elements, %											
C	Mn	Si	Cr	B	Ti	V	Al	Ni	W	S	P
0,03	0,17	0,19	13,0	2,2	3,1	0,46	0,39	0,22	-	0,005	0,02

3. Results and discussion.

As shown by X-ray and microstructural analysis, ferritic class steel 04H14T3R1F contains a large amount of boride phases of the two types: light and dark inclusions, the latter being heterophase (Fig. 1, a – c). The volume fraction of dark and light borides in the steel structure was 2.7 and 3.5% (vol.), respectively. Large borides were brittlely destroyed during hot deformation; due to the localization of stresses near inclusions, microcracks arose and the destruction of borides was facilitated [6-13].



a b c d

Fig. 1. *Boride inclusions in steel 04H14T3R1F after deformation at 1100 (a, b, c) and 1150°C (d): a - x1000, b - x6500, c - x200, d - x2500.*

The content of elements in borides of various types and in the ferrite matrix after deformation at temperatures of 1100, 1150°C was analyzed. Dark borides are heterophase inclusions of complex composition, their base is a complex boride $(Ti,Fe,Cr,V)_2B$, and the shell is a boride $(Ti,Cr,V)_2B$, in which there is a phase containing V. Analysis of the content of alloying elements in light borides at a temperature of 1100 °C showed that they are inclusions based on Fe, Cr - $(Fe,Cr)_2B$. When the deformation temperature increases to 1150°C, a change in the composition, structure and shape of borides occurs, which is associated with the diffusion redistribution of chemical elements in the borides. Analysis of the Fe-B diagram showed that borides are formed as a result of eutectic transformation, which is important for understanding their behavior under high-temperature thermal and deformation effects.

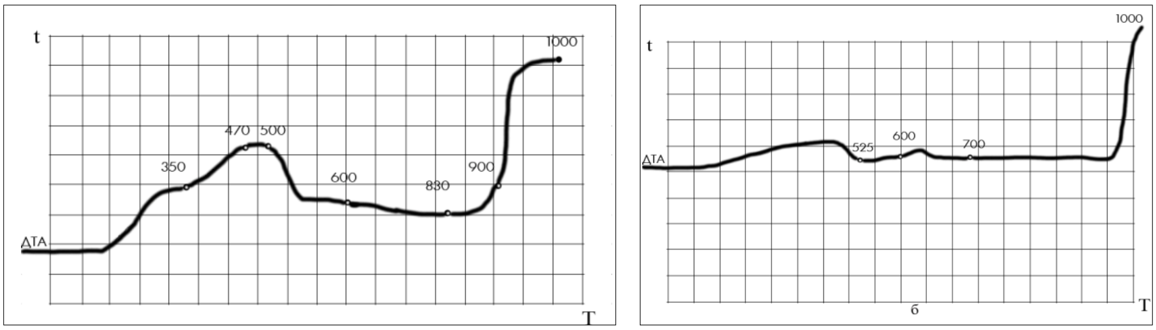
During deformation at 1150°C, dynamic diffusion interaction of borides with the ferrite matrix occurs, leading to their diffusion fragmentation, dissolution and release of new dispersed inclusions (Fig. 1, d). The mechanism of diffusion fragmentation of borides is described in [6]. The matrix of steel 04H14T3R1F is an alloyed ferrite containing 83% Fe, 14.8...18.8% Cr, 0.4...0.8% Ti, 0.3...0.38% Ni. The microhardness values of borides and the matrix indicate the development of strengthening of the ferrite matrix and borides under thermal deformation (table. 2), which should cause an increase in the strength characteristics of steel and its deformability.

Thus, in steel 04H14T3R1F, boride phases (inclusions) with wide areas of homogeneity were identified. It is known that the size of boron atoms allows, along with interatomic metal-metal and metal-nonmetal bonds, the formation of direct boron-boron bonds [3], which complicate the processes of electronic and diffusion interaction of atoms in complex borides. Analysis of the Fe-B diagram showed that borides are formed as a result of eutectic transformation, which is important for understanding their behavior under high-temperature thermal and deformation effects.

Table 2. *Size of borides D_b, microhardness of borides $H\mu^b$ and matrix $H\mu^m$ of steel 04H14T3R1F*

Metal condition	Borides based Fe, Cr		Borides based Fe, Ti		Ferrite matrix
	D_b, μm	$H\mu^b$, MPa	D_b, μm	*$H\mu^{б}$*, MПа	$H\mu^m$, MPa
Initial workpiece	90…15	3830	55…10	5490	2630
Hot rolled pipe	45…10	5490	20…52	7860	3210

The study of thermogravimetric analysis curves indicates transformations in the boride inclusions themselves. On the DTA curves (enthalpy change curve) constructed for samples from the workpiece (Fig. 2, a) and hot-deformed steel 04H14T3R1F (Fig. 2, b), kinks of different sizes are visible at temperatures below 700 °C. At temperatures above 900 °C for both types of samples, a sharp change in the course of the DTA curves is observed. In the initial workpiece, the ferrite matrix contains borides that have not undergone the influence of thermomechanical action. In hot-deformed samples, the influence of thermal deformation treatment on the structure of boride phases is obvious. Since the steel under study is in a ferritic state throughout the entire temperature range studied, the inflections in the DTA curves are due to transformations in borides. Perhaps, at a higher deformation temperature, the boride transformation $(Ti,Fe,Cr)_2B \rightarrow (Fe,Cr)_2B$ occurs. Boride transformation is also possible during the heating process of 04H14T3R1F steel under deformation, or during cooling after deformation, which affects the properties of boride inclusions.



a b

Fig. 2. *Curves of thermogravimetric analysis of steel samples 04H14T3R1F: a – workpiece; b – hot rolled strip*

The mechanical and technological properties of 04H14T3R1F steel are largely determined by the initial structure of the pipe blank, as well as the possibilities of its transformation during the process of plastic deformation. In the samples taken from the workpiece and sleeves after the first piercing, large borides of both types are present. The structure of a hot-rolled sleeve contains lines of non-plastic borides elongated in the rolling direction and smaller in size than in the pipe blank. Subsequent rolling at temperatures of 900...950°C and a degree of deformation of 34% contributes to a more uniform distribution and production of smaller borides. Taking into account the transformation processes of borides during deformation and heating, the change in the mechanical properties of 04H14T3R1F steel in the original pipe blank, sleeve and hot-rolled pipes was analyzed. A significant change in mechanical characteristics, especially ductility, occurs during the process of piercing a pipe blank into a sleeve, where the main deformation of the metal occurs (Table 3).

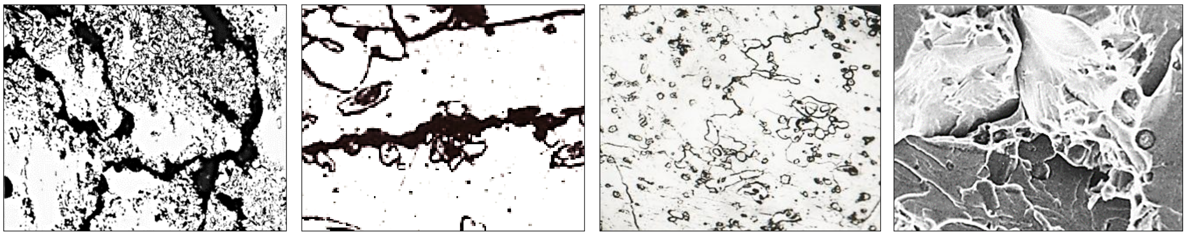
Table 3. *Mechanical characteristics of steel 04H14T3R1F*

Metal condition	ultimate strength, MPa	yield point, MPa	specific elongation, δ %	Impact strength, KCU, J/cm^2
Initial workpiece	435…452	320…372	8,2…10.3	5,1…6,2
sleeve	480…510	310…330	16,0…18,5	6,3…7,4
Hot rolled pipe	550…586	410…445	13,0…15,5	10,2…19,2

Mechanical tensile tests were carried out to determine the mechanical characteristics of 04H14T3R1F steel at elevated temperatures (Table 4). The strength properties gradually decrease with increasing test temperature, which should indicate a corresponding increase in the ductility of the steel. However, the plasticity characteristics do not demonstrate a smooth increase in their values with increasing test temperature. Obviously, this is associated both with a change in the ratio of the contribution of the processes of hot strengthening and dynamic restoration of the structure of ferrite matrix at different deformation temperatures, and with the possibility of changing the behavior of boride inclusions due to boride transformations. In addition, a drop in plastic characteristics at test temperatures of 1150...1200 °C indicates the manifestation of red brittleness due to the melting of borides (boride eutectics) (Fig. 3, a) [6,7], since the eutectic equilibrium temperature on the Fe-B diagram is 1177 °C. Thus, the test results showed that the use of elevated temperatures during piercing (1150 °C) and rolling pipes on an automatic TPA 350 installation (1050 °C) is undesirable.

Table 4. *Mechanical characteristics of steel 04H14T3R1F at elevated temperatures*

property	testing temperature, °C							
	850	900	950	1000	1050	1100	1150	1200
ultimate strength, MPa	92,3	78,1	56,4	46,2	38,5	35,6	29,3	23.2
yield point, MPa	81,2	62,4	51,3	39,3	32,4	29,6	25,4	22,4
specific elongation, δ, %	61,4	64,0	71,8	76,3	62,3	66,4	46,2	18,7
contraction ratio, ,ψ %	68,3	72,1	75,4	81,2	87,2	92,5	69,1	33,4



a b c d

Fig. 3. *Microstructure of steel 04H14T3R1F after hot deformation at temperatures of 1150 (a), 1050 (b) and 950 °C (c), as well as fracture after dynamic tests (d); a, c - x 200, b - x600, d - x500.*

It is quite obvious that the problems of unsatisfactory technological ductility are largely associated with the transformation of boride inclusions under thermal and deformation effects, which determine the behavior of the inclusions themselves at different stages of the technological process of producing pipes from steel 04H14T3R1F, as well as influencing structural changes in the ferrite matrix. In boride accumulations, due to deformation stresses, cracks appear near inclusions (Fig. 3, b), which also contributes to a decrease in the technological ductility of steel. The development of dynamic recrystallization at elevated temperatures (1000...1050 °C) and the production of ferrite grains of 1-2 points (Fig. 3, c) leads to a decrease in impact strength values to 5 J/cm2. Reducing the rolling temperature to 900...950 °C and using a degree of deformation of 34% led to the development of dynamic polygonization and recrystallization processes, which contributed to an increase in impact strength to 10...20 J/cm2. Rolling with degrees of deformation of 20 and 34% at a temperature of 850 °C contributed to the development of softening processes of the ferrite matrix, largely through the mechanism of dynamic polygonization, although signs of dynamic recrystallization were observed, with new grains forming near boride inclusions. In the structure of fractures at all pre-rolling temperatures, particles of boride inclusions, areas of brittle cleavage and secondary cracks were observed (Fig. 3, d), which indicates the localization of destruction in places where borides accumulated.

Research was carried out to determine the optimal temperatures and degrees of deformation during rolling on an automatic installation and the influence of temperature-deformation parameters of rolling on the development of destruction processes in steel 04H14T3R1F under dynamic loading. An increase of impact *strength* is achieved at a certain combination of temperature and degree of deformation: the optimal rolling temperature range is 950...900 °C, the degree of deformation is 34% (Table 5).

Table 5. *Values of impact strength of steel 04H14T3R1F after deformation at various rolling parameters*

Deformation degree, ε, %	*Impact strength*, *KCU*, J/cm^2, after deformation at temperatures				
	850 °C	900 °C	950 °C	1000 °C	1050 °C
20	4,7	7,3	5,0	5,0	5,0
34	4,4	20,8	10,4	5,0	5,0
50	4,7	5,0	5,0	5,0	5,0

4. Conclusions.

In the process of hot deformation, phase and structural transformations occur: a change in the composition of borides due to the redistribution of elements, dynamic diffusion crushing and separation of "satellite" particles, brittle destruction of borides, boride transformation, as well as melting of inclusions. Boride inclusions in the process of hot deformation are not plastic and are the sources of the appearance of cracks, which contributes to a decrease in the technological plasticity of 04H14T3R1F steel. The study of the behavior of boride inclusions during hot pressure treatment of steel and their influence on the formation of the structure of the deformed matrix, the development of destruction near the inclusions, as well as the mechanical properties made it possible to determine the treatment regimes (temperature and degree of deformation) that contribute to obtaining an optimal structure that provides increased impact *strength* of 04H14T3R1F steel.

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