

# Analysis of Nonmetallic Inclusions Distribution and Microstructure in Centrifugally Cast Steel Pipes

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**Abstract.** The type and distribution of nonmetallic inclusions determined by the qualitative and quantitative analysis and analysis of the microstructure are presented in this paper. Investigation was performed on centrifugally cast steel pipes of different diameters and wall thicknesses. Pipes were made of the CrMo and CrMoV steels. Results of the nonmetallic inclusions and microstructural analysis show that the more intensive inhomogeneity is found in centrifugally cast pipes in the radial than in the axial direction.

## Introduction

Numerous results of nonmetallic inclusions and microstructure analysis for stationary casting are presented in the literature. For the centrifugal casting, the problem of nonmetallic inclusions and microstructure distribution is more complex. The reason for this is the effect of centrifugal force. The action of the centrifugal force is higher than the force of gravity and, as a consequence, the higher density of solidified metal was obtained. The technological parameters, such as the number of the mould rotations -  $n$ , the casting temperature -  $T_1$  and the casting speed -  $v_1$ , have the crucial influence on the final product properties, beside the chemical composition and the dimensions of casting [1-3]. The number of the mould rotations shows the highest influence in the radial direction, which is very important for the formation of microstructure and the quality of casting. Due to the difference in density of nonmetallic inclusions and liquid metal and under the influence of centrifugal force, segregation is formed in the radial direction of the solidified metal. At the horizontal centrifugal casting, a characteristic segregation appears radially, especially in the case of the steel pipes castings. The analysed literature indicates that there is no significant segregation in the direction of the rotation axis [1-4].

In this paper, the type and distribution of nonmetallic inclusions are determined by the qualitative and quantitative analysis, whereas microstructural characteristics are analysed by optical microscopy. These data are of crucial interest for centrifugally cast steel pipes quality, especially if the cast pipes are used as the pre-forms for hot working.

## Experimental

The tubes have been made of CrMo and CrMoV steels of the chemical composition which is given in Table 1. Steels were produced in the medium-frequency induction furnace of 1000kg/1MW. The tube casting was produced on the centrifugal machines with horizontal rotation axis of steel moulds, at the conditions given in Table 2.

The tubes C1 and C2 made of CrMo steel were cast in the steel mould of dimensions  $\phi 264/\phi 182 \times 2100\text{mm}$ , and the tube A of CrMoV steel was cast in the steel mould of dimensions  $\phi 260/\phi 138 \times 1405\text{mm}$ . Before casting, the preheated moulds were dry-coated, the coating consists of the fine granulated  $\text{Al}_2\text{O}_3$  and formaldehyde pitch ("rezofen").

Table 1 *Chemical Compositions of CrMo and CrMoV Casting Steels*

| Steel Mark | Chemical composition, mass. % |      |      |       |       |      |      |      |       |       |       |       |       |                     |
|------------|-------------------------------|------|------|-------|-------|------|------|------|-------|-------|-------|-------|-------|---------------------|
|            | C                             | Mn   | Si   | S     | P     | Cr   | Mo   | V    | Ni    | Cu    | Al    | O     | N     | Fe <sub>rest.</sub> |
| CrMo       | 0.20                          | 0.60 | 0.44 | 0.028 | 0.032 | 1.25 | 0.15 |      | 0.160 | 0.140 | 0.080 | 0.038 | 0.014 | 96.868              |
| CrMoV      | 0.40                          | 0.70 | 0.42 | 0.022 | 0.020 | 2.70 | 0.20 | 0.10 | 0.092 | 0.097 | 0.075 | 0.037 | 0.020 | 95.117              |

Chemical compositions of the castings were determined by the spectrometric method using the quantometer. The chemical compositions of the nonmetallic inclusions in samples were analysed by energy dispersive spectroscopy (EDS) on scanning electron microscope (SEM) at the

Table 2 *The Casting Conditions of the Centrifugally Cast CrMo and CrMoV Steel Pipes*

| Steel Mark | Casting Mark | T <sub>k</sub> <sup>*</sup> (K) | T <sub>1</sub> (K) | τ <sub>1</sub> (sec) | τ <sub>h</sub> (sec) | n <sub>k</sub> (o/min) | k   | d <sub>s</sub> (mm) | δ (mm) | l (mm) | δ <sub>k</sub> /δ |
|------------|--------------|---------------------------------|--------------------|----------------------|----------------------|------------------------|-----|---------------------|--------|--------|-------------------|
| CrMo       | C1           | 423                             | 1863               | 35                   | 700                  | 1562                   | 102 | 176                 | 50     | 2000   | 0.82              |
| CrMo       | C2           | 423                             | 1863               | 35                   | 600                  | 1315                   | 101 | 172                 | 33     | 2000   | 1.24              |
| CrMoV      | A            | 423                             | 1873               | 25                   | 420                  | 1012                   | 42  | 128                 | 25     | 1400   | 2.44              |

\*where: T<sub>k</sub> - Mould temperature [K]; T<sub>1</sub> - Pouring temperature [K]; τ<sub>1</sub> - Pouring time [sec]; τ<sub>h</sub> - Cooling time [sec]; n<sub>k</sub> - Mould rotation speed [r/min]; k = F<sub>c</sub>/F<sub>g</sub> - Gravity coefficient; F<sub>c</sub> - Centrifugal force [N]; F<sub>g</sub> - Gravity force [N]; d<sub>s</sub> - Outside tube diameter [mm]; δ - Tube wall thickness [mm]; δ<sub>k</sub> - Mould wall thickness [mm]; l - Tube length [mm]

magnification of 503 times. The measurements were performed across the the tube walls in n – zones, located from the tube periphery (from zone 1) to the centre (to the inside region) (Fig. 1). The surface of each zone was 9 mm<sup>2</sup>. The samples were prepared by mechanical grinding and polishing.

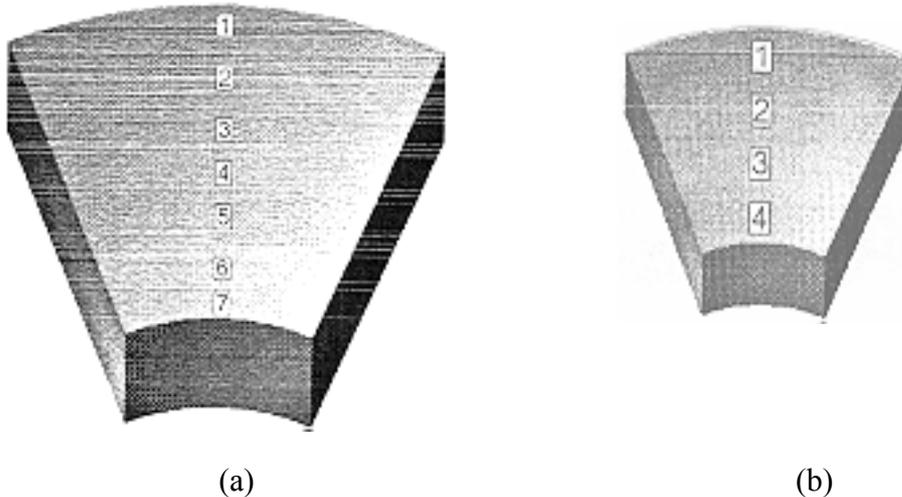


Fig. 1. The position of measured zones in the radial direction: a) steel C1; b) steel A

The analysis of distributions of the nonmetallic inclusions were determined by automatic and semi – automatic quantitative area method on polished samples (Fig. 1) by the image analyzer on 100 consecutive measuring areas of each zone. The microstructure of the pipe specimens was analyzed by optical microscope at the magnification of 100 and 125 times. The samples were prepared by mechanical grinding and polishing and then were etched in 3% Nital.

## Results and Discussion

The qualitative analysis of nonmetallic inclusions of the centrifugally cast pipes is presented in Table 3. Nonmetallic inclusions which were identified in the radial direction across the wall thickness of the investigated pipes (C1, C2 and A) were of the following shapes:

- Sulphides - as globular, irregular, X and colonial shape;
- Oxisulphides - as globular, irregular and longer (ellipsoid) shape;
- Oxide - as globular and irregular shape.

The nonmetallic inclusions shape of sulphides, oxides and oxisulphides verified by SEM analysis in the pipes C1 and A are presented in Fig. 2.

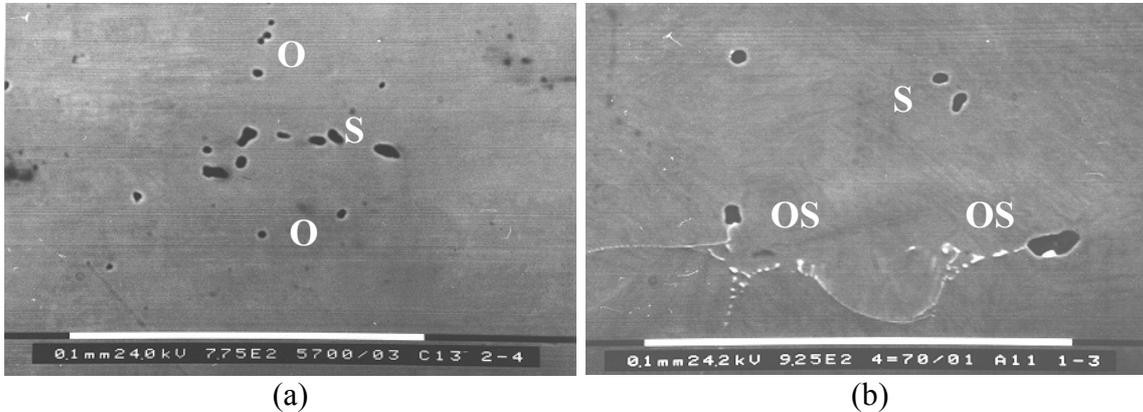


Fig. 2. SEM micrograph. The shapes of nonmetallic inclusions: a) oxides (O) and sulfides (S) of nonmetallic inclusions in the pipe C1, zone 1; b) sulfides (S) and oxisulphides (OS) of nonmetallic inclusions in the pipe A, zone 1.

Table 3. Chemical Composition and Shape of Nonmetallic Inclusions in the Centrifugally Cast Pipes

| Type inclusion  | Chemical composition, in mass % |       |       |       |       |       |       |       | Shape inclusion |
|---|---------------------------------|-------|-------|-------|-------|-------|-------|-------|-----------------|
|   | Cr                              | Mo    | Si    | Mn    | S     | Al    | V     | Fe    |                 |
| MnSFeS·CrS  | 3.76                            | 22.60 | /     | 30.61 | 7.90  | 0.34  | 0.15  | 34.26 | Globular        |
|   | 5.03                            | 15.42 | /     | 46.52 | 13.14 | 0.77  | 0.09  | 19.03 |                 |
|   | 11.71                           | 11.13 | /     | 36.70 | 20.05 | 0.26  | 0.25  | 19.76 | X               |
|   | 3.91                            | 5.13  | 0.16  | 15.31 | 5.14  | 0.31  | 0.55  | 69.02 | Irregular       |
|   | 5.72                            | /     | /     | 24.56 | 10.14 | /     | 0.25  | 59.12 |                 |
|   | 2.22                            | 35.91 | 0.46  | 12.85 | 25.71 | 0.84  | 1.85  | 20.16 | Colonial        |
| MnSFeS·CrS·Al <sub>2</sub> O <sub>3</sub> ·FeO·SiO <sub>2</sub> | 1.72                            | 22.12 | 0.09  | 5.44  | 2.27  | 1.94  | 0.13  | 66.20 | Globular        |
|   | 3.91                            | 16.45 | 0.20  | 41.45 | 18.31 | 9.52  | /     | 10.17 |                 |
|   | 2.82                            | /     | 0.61  | 12.43 | 2.63  | 19.56 | 0.41  | 61.37 |                 |
|   | 3.01                            | 12.77 | 0.51  | 8.94  | 2.52  | 9.44  | 0.15  | 62.47 |                 |
|   | 3.21                            | 10.30 | 0.41  | 13.83 | 7.55  | 22.08 | 0.21  | 42.45 | Ellipsoid       |
|   | 3.19                            | 6.50  | /     | 19.48 | 3.45  | 25.79 | /     | 41.29 |                 |
|   | 3.11                            | 11.73 | 0.50  | 41.64 | 24.95 | 10.48 | /     | 7.59  | Irregular       |
| 8.10  | 12.04                           | 0.10  | 23.00 | 5.16  | 22.05 | 0.10  | 29.36 |       |                 |
| FeO·SiO <sub>2</sub>  | 1.21                            | /     | 37.70 | 0.89  | 0.14  | 0.32  | /     | 59.74 | Globular        |
| FeO·SiO <sub>2</sub>  | 0.83                            | /     | 63.60 | 0.60  | /     | /     | 0.32  | 34.66 | Irregular       |
| FeO·SiO <sub>2</sub>  | 1.94                            | 14.35 | 69.12 | /     | /     | 1.05  | /     | 13.53 | Globular        |
| FeO·Al <sub>2</sub> O <sub>3</sub> ·SiO <sub>2</sub>            | 0.95                            | 11.18 | 10.38 | 0.27  | /     | 14.42 | 0.16  | 62.64 |                 |
| FeO·MnO·Al <sub>2</sub> O <sub>3</sub>                          | 1.06                            | 10.42 | /     | 1.73  | 0.11  | 28.02 | /     | 58.67 | Globular        |

The results of the EDS analysis of the pipes have shown that the following types of nonmetallic inclusions are detected: sulphides as MnSFeS·CrS; oxisulphides as MnSFeS·CrS·Al<sub>2</sub>O<sub>3</sub>·FeO·SiO<sub>2</sub>; oxides as FeO·SiO<sub>2</sub>, FeO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> and FeO·MnO·Al<sub>2</sub>O<sub>3</sub>.

The presented results have confirmed that the nonmetallic inclusions identified in cast pipes as sulphides and oxisulphides were of globular and irregular shapes. They were mostly distributed at

grain boundaries or in the form of fine eutectics (white spots at grain boundaries as presented in Fig. 2) and could be classified as the first and the second type of inclusions according to Simsu and Dehlu [5].

On the other side, inclusions identified as oxides are present in globular and irregular shape. Their composition probably correspond to complex compounds as  $\text{FeO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ ;  $\text{FeO}\cdot\text{MnO}\cdot\text{Al}_2\text{O}_3$  and  $\text{FeO}\cdot\text{SiO}_2$  [6, 7]

The quantitative analysis and distribution of nonmetallic inclusions in the radial direction across the wall thickness, in the case of the pipes C1, are presented in Fig. 3.

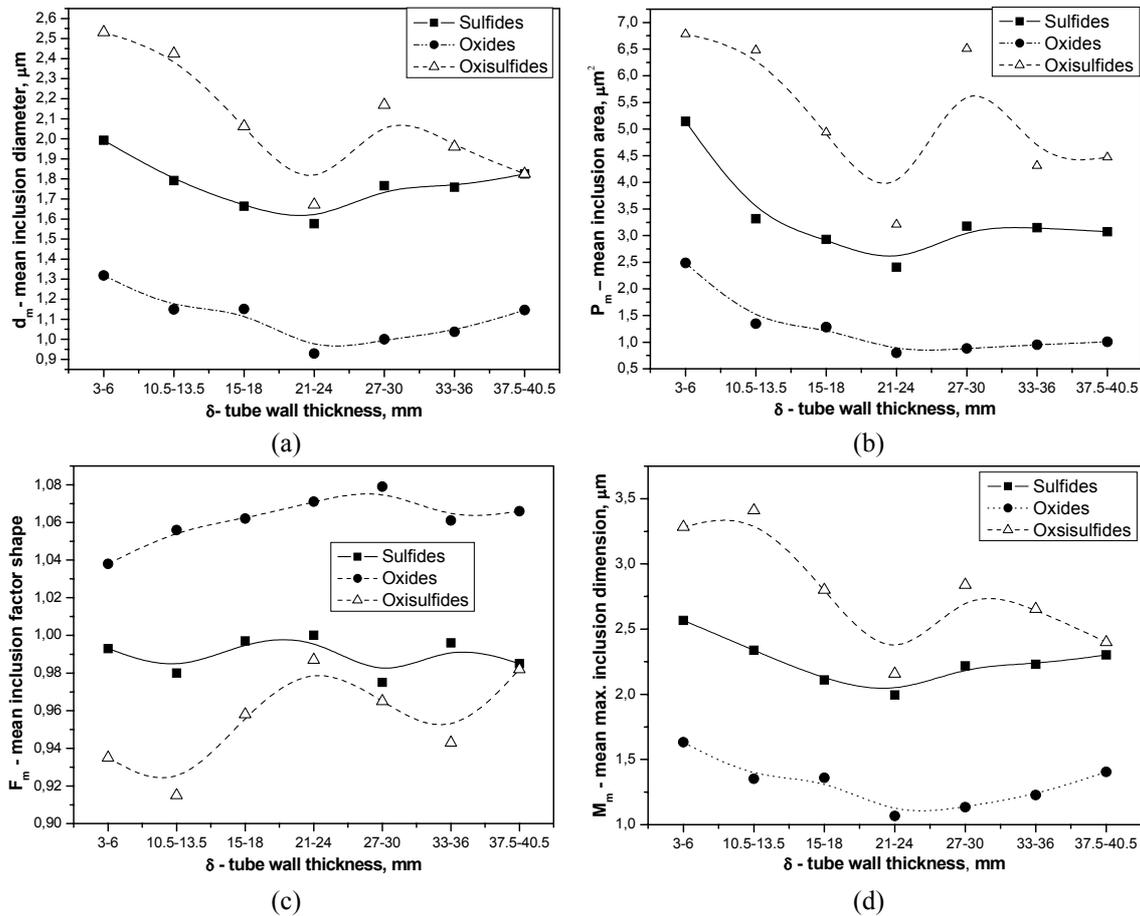


Fig. 3. The distribution of nonmetallic inclusions in the radial direction of the centrifugally cast steel pipe C1: a)  $d_m$  - mean inclusion diameter,  $\mu\text{m}$ ; b)  $P_m$  - mean inclusion area,  $\mu\text{m}^2$ ; c)  $F_m$  - mean inclusion factor shape; d)  $M_m$  - mean max. inclusion dimension,  $\mu\text{m}$ .

Radial distribution of nonmetallic inclusions demonstrates the occurrence of segregations in centrifugally cast pipe C1. The distribution curves of radial segregation of nonmetallic inclusions as:  $d_m$  - mean inclusion diameter (Fig. 3a),  $P_m$  - mean inclusion area (Fig. 3b) and  $M_m$  - mean maximal inclusion dimension (Fig. 3d) are similar. It is evident that the size, area and maximal dimension of inclusions are higher in the peripheral zone (zone of contact between the mould and the pipe) of the casting and in the inside zone (the zone of contact between the casting and the cooling air) than in the middle zone of the pipe. In the inside zone, up to approximately 10 - 20 mm of the wall thickness, the size of inclusions decreases. At the higher radial distances from inner wall, the size of the nonmetallic inclusions is either constant or increases. The distribution of the oxisulphides shows a scatter of the results. The shape of the sulphides inclusion is approximately globular across the whole wall thickness of the pipes as presented in Fig. 3c. To the contrary, the shape of the oxides and oxisulphides is not so regular. Near the casting surface, which is in contact with the mould, the oxides inclusions are more globular than the oxisulphides inclusions. In the inside zone (the zone of contact of the casting and the air) this phenomenon is reversed.

The radial segregation of nonmetallic inclusions of the centrifugally cast steel pipe C1 is the result of directional crystallization, the highest rotation speed of the mould and the lowest ratio of mould/wall thickness and the tube/wall thickness  $\delta_k/\delta=0,82$  (Table 2). The maximal measured size of inclusions detected in the investigated cast pipes  $\leq 15 \mu\text{m}$ , which is smaller than the values presented in the literature ( $< 20 \mu\text{m}$ ) [2, 3, 8] and rather smaller than was results obtained by the theoretical calculations ( $< 26 \mu\text{m}$ ) [2].

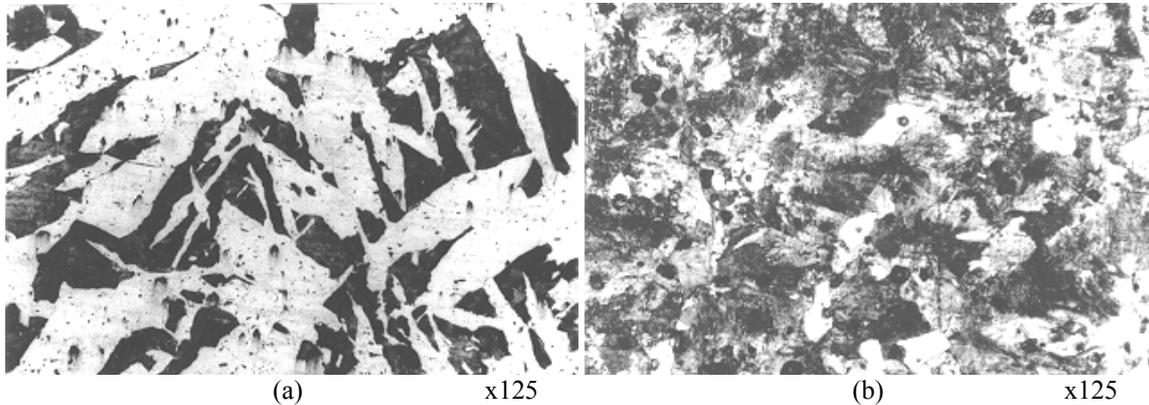


Fig. 4. Optical micrographs. The radial microstructure of the centrifugally cast steel pipe C1, etched by the 3% Nital: a) the 1-3 mm deep peripheral layer, b) the deep layer 40-46 mm inside tube region

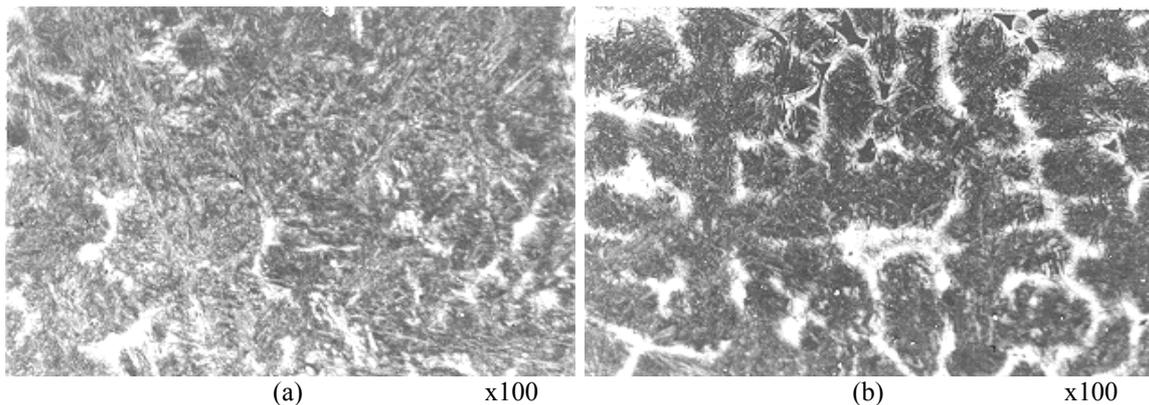


Fig. 5. Optical micrographs. The radial microstructure of the centrifugally cast steel tube A, etched by the 3% Nital: a) the 1-3 mm deep peripheral layer, b) the deep layer 17-23 mm inside tube region.

The general microstructure of the pipe C1 castings consists of ferrite and dispersed pearlite as presented in Fig. 4. Within the ferrite grains and at their boundaries of irregular shape, the fine precipitates are observed. The pearlite and ferrite grains are separated with the sharp-angle boundaries. In addition, the pearlite areas are of irregular shape and size. Generally, the microstructure in the zone which is in contact with the mould is different in morphology and the fraction of microconstituents (Fig. 4a) compared to the middle zone and to the zone from the inside region of the pipe (Fig. 4b), i.e. the structure is more dispersed in the middle and in the inside region of the C1 pipe. Such a structure is also the result of the same parameters that influenced the radial segregation of nonmetallic inclusions.

The microstructure of the castings of the pipe C2 and A consists of the ferrite and bainite and the martensite and bainite, respectively, and is distinguished by the more fine-grained structure (Fig. 5a and b). This microstructure which is quite different compared to the microstructure of C1 pipe (Fig. 4) originates from more intensive cooling conditions of the pipes C2 and A. This higher cooling rate is a consequence of higher ratio of the mould wall thickness and the pipe thickness  $\delta_k/\delta = 2.44$  compared to the pipe C1 (see Table 2).

## Summary

Distribution of nonmetallic inclusions and microstructure in centrifugally cast pipes of various wall thicknesses made of CrMo and CrMoV steel are presented in this paper. The complex inclusions such as sulphides  $\text{MnS}\cdot\text{FeS}\cdot\text{CrS}$ , oxisulphides  $\text{MnS}\cdot\text{FeS}\cdot\text{CrS}\cdot\text{Al}_2\text{O}_3\cdot\text{FeO}\cdot\text{SiO}_2$ , oxides  $\text{FeO}\cdot\text{SiO}_2$ ,  $\text{FeO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$  and  $\text{FeO}\cdot\text{MnO}\cdot\text{Al}_2\text{O}_3$  with globular and irregular shape were present in pipes. In centrifugal cast pipes the more intensive growth of radial nonmetallic inclusions segregation is observed than in the axial direction. The shape of sulphide inclusions is approximately globular across the whole walls of the pipes. However, oxide inclusions are more globular than oxisulphide inclusions near the casting surface. In the inside zone (the zone of contact of the casting and the air) this phenomenon is reversed.

The microstructure of the pipe casting of the higher wall thickness made of CrMo steel consists of ferrite and dispersed perlite. In the smaller wall thickness pipes made of CrMo and CrMoV steel the ferrite and bainite and the martensite and bainite are present, respectively. The microstructure of these pipes is characterized by the more fine-grained structure in the peripheral zones than in the pipe of larger diameters and wall thickness.

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