Ways to Improve the Quality of Die Steel 5KhNM

S V Efimov\textsuperscript{1}, O Yu Malykhina\textsuperscript{1}, A G Pavlova\textsuperscript{1}, V G Milyuts\textsuperscript{2}, V V Tsukanov\textsuperscript{2} and V V Vikharev\textsuperscript{2}

\textsuperscript{1}OMZ-Special Steels LLC, St. Petersburg, RU-196650, Russia  
\textsuperscript{2}The Federal State Unitary Enterprise Central Research Institute of Structural Materials “Prometey” 49 Shpalernaya Street, St. Petersburg, RU-191015, Russia

E-mail: Semen.Efimov1@omzglobal.com

Abstract. There was performed an analysis of influence of the deoxidation technology, hydrogen content and high concentration of titanium in steel 5KhNM (Rus. “5XHM”) on quality of die blanks, evaluated based on the results of the ultrasonic test. The fractographic examinations of fractures and the X-ray microprobe analysis of chemical composition of non-metallic inclusions were conducted, the evaluation of macro- and micro-structure of a die blank with high titanium content was performed. It is demonstrated that defects of dies from steel 5KhNM (Rus. “5XHM”) are cracks from merged flakes and micro-flakes; in most cases large concentrations of sulphides appeared to be hydrogen collectors for formation of flakes and micro-flakes.

1. Introduction

Growth of unit capacity and technical characteristics of nuclear power plants, large power plants and chemical plants conditions the increase of weight and size of equipment parts. Die plates manufactured from large forgings of alloyed steels being in stable and steady demand on the domestic and international markets are used for manufacture of such products.

Manufacture of high-quality large forgings from forging ingots of large weight is by far a complex technical task, which is attributable to strong development of segregation processes, non-uniform distribution of non-metallic inclusions, flakes formation, physical heterogeneity inside an ingot and other deficiencies related to the process of solidification of large amount of metal. Moreover, increase of ingot weight results in enhanced development of defects.

One of common internal defects of forgings is flakes, which significantly deteriorate strength characteristics and plasticity of steel and structural strength of products made of such steels. According to classification of internal defects, suggested in paper [1], flakes are described as a defect of macrostructure in the form of thin curly cracks with a length of up to several dozens of millimetres located arbitrarily in different directions throughout the metal section except for the edge area. Flakes belong to the group of unacceptable defects.

Nowadays, it is considered proven [2, 3] that flakes are internal micro-voids filled with hydrogen. Nevertheless, so far there is no consensus on the mechanism of flakes formation, hydrogen “critical content” for their formation, and the role of nature and composition of non-metallic inclusions.
A. N. Morozov believed that the hydrogen threshold level ensuring absence of flakes in steel is 2.0 to 2.5 ppm [4]. The authors [5] also support this model of flakes formation [5].

On the other hand, papers [6-8] suggest the opinion that reduction of hydrogen content even significantly below this concentration does not guarantee absence of flakes in steel with very low sulphur content (less than 0.005%).

Therefore, known beliefs about conditions, under which flakes are formed, require clarification due to technology development providing extra low content of hydrogen, additives and non-metallic inclusions.

The authors of papers [9–13] expressed the hypothesis that flake germs are manganese sulphides, and it is related to their certain peculiarities, that is, they precipitate last of all during steel crystallisation, they fall into the shrinkage area, and their thermal expansion coefficient is much larger than the iron’s one. When the ingot is cooling, manganese sulphides are decreasing in size more than the surrounding metal, which results in formations of cracks between manganese sulphides and metal, and as a consequence, these cracks presumably are the centres of flakes origination. The available empirical evidence indicates that manganese sulphides being “hydrogen traps” influence flakes origination. The amount of manganese sulphide particles decreases at very low sulphur concentration in steel, flakes are formed extremely intensively and it leads to noticeable growth of rejects because of flakes. This is due to the fact that because of low amount of manganese sulphides, the hydrogen concentration increases being attributable to each inclusion. This raises hydrogen partial pressure and enhances the possibility of cracks generation and development [6, 14].

Because of existing cases of titanium content in die steel 5KhNM (Rus. “5XHM”) in the amount of 0.01–0.03% and more, which is significantly higher than the typical level, it is reasonable to view its influence on steel quality, since it is known that titanium content of more than 0.03% can result in deterioration of the quality of steel for castings because of possible formation of large sulphides and nitrides along the boundaries of grains [15, 16].

The analysis of literature sources, including those devoted to examination of quality of large forgings from alloyed steels, indicates that they almost do not contain any information on technological special aspects of manufacturing of forged products from large weight ingots.

At the same time, there remains an urgent need to develop optimal technological manufacturing methods preventing defects formation in large forgings.

This paper aims to study the influence of process parameters of melting and chemical composition on quality of forgings from steel 5KhNM (Rus. “5XHM”).

2. Materials and Research Method

Stamp steel of 5KhNM (Rus. “5XHM”) grade was smelted in the electric arc furnace EAF-120, and the semi-finished product was treated in the ladle refining installation.

The steel was teemed into forging ingots by the siphon method with protection of the metal stream from secondary oxidation by argon.

Two schemes of metal deoxidation during refining were used at steel smelting:

- Scheme 1. Without aluminium (8 heats);
- Scheme 2. With addition of aluminium before vacuum degassing (9 heats).

For trial heats, aluminium was introduced as flux-cored wire or as the aluminium chop over the slag.

The analysis of results of forgings production was carried out based on heat card data, ultrasonic test (UST) cards, forging data and heat treatment data.

There was determined the influence of the deoxidation scheme and hydrogen content in metal after vacuum treatment on quality of forgings.

The results of production of forgings from three other heats with titanium content in steel from 0.03 to 0.055%, which is 10 to 15 times higher than the regular level of its concentration in steel 5KhNM (Rus. “5XHM”), were analysed.
In view of high level of rejection of forgings from these heats, a sample taken from one of the rejected forgings from the heat under the reference number 3 was analysed in order to determine the type of indications determined during the ultrasonic testing.

Two templates were cut in longitudinal and transverse directions relative to the billet longitudinal axis. Then macro- and microsections were made from these templates, as well as samples for three-point bending were made out of the template in the longitudinal direction.

Macrostructure was determined by the chemical etching method in 15% water solution of ammonium persulphate.

Metal impurity by non-metallic inclusions was evaluated by comparison with GOST 1778-70 scales increased hundred times.

The fractographic examinations of fractures and the X-ray microprobe analysis of chemical composition of non-metallic inclusions were conducted by a scanning electron microscope and an analysing add-on device. Fractures were received by the three-point bending method by disclosure of defects revealed at macrosections. The X-ray microprobe analysis of non-metallic inclusions was performed at microsections and from fractures.

3. Study Results

3.1. The Influence of Smelting Technology and Steel Chemical Composition on Quality of Forgings

The results of the ultrasonic test of forgings made from metal smelted according to two deoxidation schemes being studied are provided in Table 1.

| Technology Scheme | Deoxidation | Number of Heats, pcs. | Content in Steel, % | Number of Ingots, pc/t | Forgings Rejection Rate (UST), % |
|-------------------|-------------|-----------------------|---------------------|------------------------|-----------------|
| Drivers           | Ca          |                        |                     |                        | 6.8             |
| 1                 | Al, Ca      | 8                     | 0.0055              | 0.004                  | 17/600.35       |
| 2                 | +           | 9                     | 0.0055              | 0.004                  | 22/771.95       |

Comparison of the results of die steel production with aluminium and without it did not reveal any noticeable advantage of one way of metal deoxidation over another one.

Taking into account quite similar values of rejection rate of blanks for dies manufactured according to both ways of deoxidation, further study was performed using data from all heats without separation into methods.

The amount of rejected forgings, according to the UST, depends to a large extent on hydrogen content in metal after vacuum treatment. When hydrogen concentration was 0.8 ppm and less, defects in forgings were not found, and when this parameter was 0.9 ppm and higher, the rejection rate for forgings was 16.9% (Table 2).

Data from Table 2 suggest that the critical hydrogen content, ensuring absence of flakes in 5KhNM (Rus. “5XHM”) steel forgings is about 0.5 ppm.

This conclusion is supported by the results of the previously performed research on combined influence of hydrogen and sulphur content in metal on flakes formation in large forgings [6].

At the same time, the experiments demonstrated that the actually achievable level of hydrogen concentration in the entire scope of manufactured die steel is 0.8 ppm and less under conditions of certain production.
Table 2. Influence of hydrogen content in steel 5KhNM (Rus. “5ХНМ”) on quality of forgings.

| Hydrogen Content in Metal After Vacuum Degassing, ppm | Number of Heats, pcs. | Number of Ingots, pc/t | Forgings Rejection Rate (UST), % |
|------------------------------------------------------|-----------------------|------------------------|---------------------------------|
| 0.5                                                  | 10                    | 22/798.3               | 0                               |
| 0.6 - 0.8                                            | 1                     | 2/47.9                 | 0                               |
| 0.9 and more                                         | 6                     | 15/526.1               | 16.9                            |

The analysis of the results of production of die steel 5KhNM (Rus. “5ХНМ”) with the titanium content of 0.03 to 0.055% (regular level of titanium concentration is 10 to 15 times lower and equals to 0.003 to 0.004%) demonstrated deterioration of forgings quality in this case. 40% of forgings produced from metal from three heats with higher content of titanium suffered defects, determined by the UST (Table 3).

Table 3. Results of UST of forgings manufactured from steel 5KhNM (Rus. “5ХНМ”) with titanium content of 0.03 to 0.055%.

| Heat Reference Number | Deoxidation | Content in Steel, % | Number of Ingots, pc. | Forgings Rejection Rate (UST), pc./% |
|-----------------------|-------------|---------------------|-----------------------|--------------------------------------|
|                        | Al          | Ca                  | S, %                  | Ti, %                  | H2, ppm                |                               |
| 1                     | -           | -                   | 0.008                 | 0.03                   | 1.1                    | 2                             | -                               |
| 2                     | -           | -                   | 0.002                 | 0.03                   | 0.5                    | 3                             | 1/33.3                          |
| 3                     | -           | -                   | 0.005                 | 0.055                  | 1.4                    | 5                             | 3/60                            |
| TOTAL                 | -           | -                   | -                     | -                      | -                      | 10                            | 4/40                            |

The possible reason of high titanium content, not typical for steel 5KhNM (Rus. “5ХНМ”), is the fact that steel from these heats was subject to ladle treatment right after production of titanium-stabilized chrome nickel steel in these ladles. Therefore, it is recommended not to treat steel in ladles after production of chrome nickel steel with titanium in them in order to improve the quality of forgings from die steel.

3.1. Examination of Forging Steel with Higher Content of Titanium and Defects Determined by the UST

3.1.1. Macrostructure Examination. Flake-type thin curly cracks with the length of 3 to 12 mm parallel to the longitudinal axis of the blank (Figure 1) were observed after etching when studying the macrosections.
Figure 1. Metal macrostructure (longitudinal direction)  
(a) general view of macrosection; (b), (c) micro-cracks (pointed by the arrows).

3.1.1. Microstructure Examination and X-Ray Microprobe Analysis. Examination of longitudinal and transversal microsections without etching confirmed presence of flake-type thin curly cracks formed in the area of large concentrations of non-metallic inclusions of yellow-pink (predominantly) and grey colour (Figure 2). The X-ray microprobe analysis demonstrated that yellow-pink inclusions are titanium sulphides containing carbon, and grey inclusions are manganese sulphides.

Figure 2. Flake-type curly cracks formed in the area of large concentration of sulphides, the total length of the crack is ~1.5 mm, longitudinal direction: (a) general view, × 100; (b), (c) – fragments of the crack (pointed by arrows), high-power image, × 1,000.
Impurity of sample steel by sulphides corresponds to 4 points, by point-like nitrides to 4 points, and brittle silicates to 2 points of GOST 1778-70 scale (Figure 3). The nitrides are titanium nitrides/carbonitrides.

Steel impurity by titanium sulphides and titanium nitrides/carbonitrides, not typical for steel 5KhNM (Rus. “5XHM”), is significant.

Figure 3. Non-metallic inclusions in sample steel, x100:
(a) point-type nitrides, transversal direction;
(b) sulphides and nitrides, transversal direction;
(c) brittle silicates and nitrides, longitudinal direction.

3.1.4. Fractographic Examination and X-Ray Microprobe Analysis The examination indicted that defects represent cracks formed from merged flakes/micro-flakes. Large concentrations of titanium sulphides also containing carbon (Figure 4) served as the hydrogen collector for them.

Figure 4. Photomicrographs of fracture surfaces.
(a), (b) – hydrogen collector – large concentrations of non-metallic inclusions.
4. Conclusion
The influence of the deoxidation technology, hydrogen content and higher concentration of titanium in steel on flakes formation in forgings from die steel 5KhNM (Rus. “5XHM”) was studied.

It is demonstrated that critical hydrogen content in steel, which provides absence of flakes, is 0.5 ppm.

The risk of flakes formation significantly increases when titanium content in steel is 0.03% and more. In this case, large concentrations of titanium sulphides appear to be hydrogen collectors.

It is recommended not to treat die steel 5KhNM (Rus. “5XHM”) in ladles right after manufacturing of chrome nickel steel stabilized by titanium in them, and vacuum degassing shall be performed providing hydrogen content in steel not greater than 0.8 ppm.

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