# APPLICATION OF THE BAS EN ISO 1463:2022 STANDARD IN THE CHARACTERIZATION OF PIPE MATERIALS FOR THERMAL ENERGY PLANTS

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**ABSTRACT**
*Long-term exposure of superheater and steam intermediate superheater pipe materials to high temperatures in the operating conditions that prevail in thermal power plants leads to the appearance of the oxide layer.Oxide layer forms on the inner wall of the pipe, which acts as a heat insulator and reduces heat transfer through the pipe wall.The action of the oxide layer as an insulator and limiting the transfer of heat to water vapor inside the pipe leads to overheating of the pipe wall.This paper presents the measurement of the thickness of the oxide layer on the outer and inner side of the pipeusing the standard method BAS EN ISO 1463:2022 with a presentation of the measurement results with pronounced measurement uncertainty.*

## 1. INTRODUCTION
The high temperatures and pressures that exist in the components of thermal power plants - pipes of superheaters and steam reheaters, lead to changes in the structure of the material as well as the appearance of an oxide layer.The appearance of overheating of the material due to the formation of an oxide layer on the inside of the pipe wall can have a significant impact on the service life of the component.

## 2. OXIDE LAYER
The oxide layer that appears on the pipe walls of superheaters and steam reheaters in thermal power plants acts as an insulator and leads to overheating of the pipe wall. The presence of deposits of oxide layers on the pipe walls increases the temperature of the metal, which accelerates the creep mechanism and thus reduces the service life of the material [1]. On the inner surface of a pipe made of low-alloy steel (up to 3% Cr), which is exposed to steam generated in boilers, an oxide layer is formed at different levels.When the temperature of the metal is below 560°C and there is a high partial pressure of oxygen, a layer of magnetite ($Fe_3O_4$) and hematite ($Fe_2O_3$) appears.At higher temperatures, an additional layer of wustite (FeO) may appear.If the steel has more alloying elements, spinel oxide $(Fe, Cr, Mo)_3O_4$, can form as the oxide layer grows in the direction of the pipe wall.These oxides are formed according to the following reactions [1,2]:

*Wustite:*
$$Fe + \frac{1}{2}O_2 = FeO;$$
*Magnetite:*
$$3FeO + \frac{1}{2}O_2 = Fe_3O_4$$

*Hematite:*

$$2Fe_3O_4 + \frac{1}{2}O_2 = 3Fe_2O_3$$

Wustite is the lowest valence iron oxide that exists over a wide compositional range that does not quite include the stoichiometric composition of FeO.

Magnetite is an iron oxide of intermediate valence that has a composition close to the stoichiometric composition of $Fe_3O_4$.Magnetite is the most abundant oxide on the inner wall of superheater and steam reheater tubes operating in conventional chemical cycles.

Hematite is the highest valence iron oxide with a composition close to the stoichiometric composition of $Fe_2O_3$ [3,4].

### 2.1 Consequences of the appearance of the oxide layer

In exploitation conditions that exist in thermal power plants, the appearance of the oxide layer can result in:

a) reduction of the cross-sectional area of the pipe material - which leads to an increase in stress;
b) thermal insulation of the pipe - the oxide layer has a significant effect on the heat transfer of the pipe, while an increase in the temperature of the pipe material leads to accelerated material;
c) peeling of oxide scales - peeling of thick oxide would be useful in terms of reducing the effect of thermal insulation.However, the scaled oxide can lead to pipe overheating if trapped in the system, thereby reducing the steam flow rate within the pipe [1].

## 3. MATERIAL AND METHOD OF MEASUREMENT

For the purposes of this work, the sample of steam reheater pipe ϕ 45x6 mm, made of material 13CrMo4-4was prepared. After years of exposure to the high temperature and pressure that exist in thermal power plants, a significant presence of oxide layers on the inner and outer walls of the pipes was observed. The chemical composition of pipes according to the standard DIN 17175 [5] and chemical analysis are given in Table 1.

*Table 1. Chemical composition*

| | Mass (%) | | | | | | |
|---|---|---|---|---|---|---|---|
| | C | Si | Mn | P | S | Cr | Mo |
| Standard DIN 17175 | 0,10-0,18 | 0,10-0,35 | 0,40-0,70 | max 0,035 | max 0,035 | 0,70-1,10 | 0,45-0,65 |
| Pipe ϕ 45x6 13CrMo4-4 | 0,14 | 0,25 | 0,57 | 0,011 | 0,019 | 0,70 | 0,35 |

The cross-section of the pipe sample was prepared in accordance with standard ASTM E 3-Standard Guide for Preparation of Metallographic Specimens [6]. The cutting was carried out on a CUT machine with a diamond cutting plate with the addition of an emulsion for cooling the cutting surface. Rough preparation - wet grinding on sandpaper grades 240, 400, 600, and 1000 SiC, and fine preparation - polishing with diamond suspension grades 9 and 3 microns was carried out on a machine for automatic sample preparation.

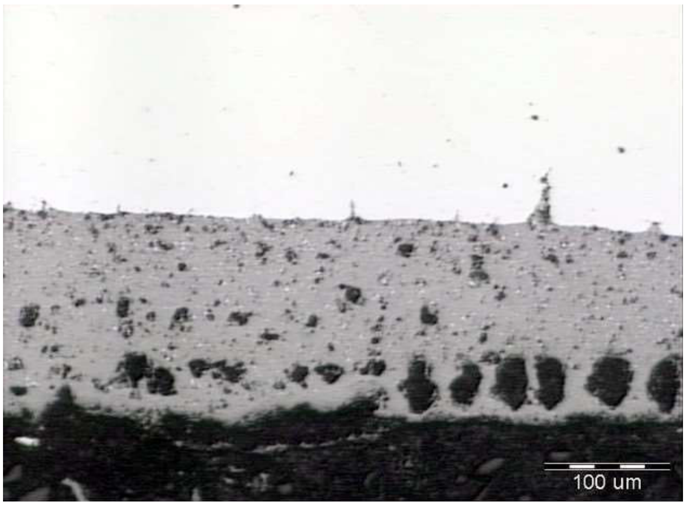
The prepared test surface was observed on a calibrated light microscope at magnifications of 50 and 100 times.

Standard BAS EN ISO 1463:2022 [7] describes, among other things, the method of measuring the thickness of oxide layers. Sample preparation - grinding, polishing and, if necessary, etching is carried out in order to obtain clearly defined layers.

### 3.1 Measurement of thickness of the oxide layer

The thickness of the oxide layer is measured using the software Analysis 5.1 with a light microscope OLYMPUS PMG3.The input to the software is calibrated using a calibrated object micrometer. The choice of magnification at which the measured oxide layer is observed is between one and a half and three times the thickness of the oxide layer. The appearance of the pipe from which the sample was taken is given in Figure 1. Representative micrographs of the oxide layer are given in Figure 2.

*Figure 1. Pipe of steam reheater, after exploitation [8]*




Uneatched x150

*Figure 2. The oxide layer, inside the wall of the pipe [8]*

The results of measuring the thickness of the oxide layer and the uncertainty of measurement of the thickness of the oxide layer are given in Table 2. The calculation of measurement uncertainty is done in accordance withthe instruction of Determination of measurement uncertainty when measuring the thickness of the metal or oxide layer UMT7-06-01 [9].

PIPE ϕ45x6 mm, material 13CrMo4-4;
Overlap factor for t-distribution (Student's distribution):
Number of degrees of freedom: f = n-1 = 5-1 = 4 and the confidence level P = 68, 27 is: t = 1,11
Measurement of the uncertainty of micrometer from calibration certificate: 2,38
Coverage factor: 2

*Table 2. Measuring the thickness of the oxide layer and uncertainty of measurement*

| No.of ofmeasur. n | Thickness layer $D_i$ (μm) | Mean value $D_{sr}$ | Standard deviation $S_D$ | Measur. uncertainty $\mu_D$ | Measur. of micrometer $\mu_C$ | Combined uncertainty $\mu_C(D)$ | Extended measure. uncertainty $\mu(D)$ |
|---|---|---|---|---|---|---|---|
| 1. | 163,2 | | | | | | |
| 2. | 166,0 | | | | | | |
| 3. | 166,9 | 165,18 | 1,883 | 1,045 | 1,19 | 1,584 | 3,2 |
| 4. | 166,7 | | | | | | |
| 5. | 163,1 | | | | | | |

The result of measuring the thickness of the oxide layer was: 165,2±3,2 microns.

## 4. CONCLUSION

The measured value of the thickness of the oxide layer on the inner wall of the pipe ϕ45x6 mm, made of material 13CrMo4-4 in the value of 165.2±3.2 μ indicates that attention should be paid to the remaining life of the pipe.Namely, the application of an oxide layer on the inner wall of the pipe for operational conditions in thermal power plants (temperatures over 450°C) can lead to degradation of the microstructure [1], because the oxide layer will act as a thermal insulator, causing local overheating of the pipe.
The BAS EN ISO 1463:2022 standard provides a good basis for measuring the thickness of the oxide layer and seeing the general picture in the characterization of pipe materials used in thermal power plants, as well as estimating the remaining life of the pipe material.

## 5. REFERENCES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