

PAPER • OPEN ACCESS

Electronic and microscopic researches of dislocation structure of metal near crack top

To cite this article: O A Nasibullina and A S Tyusenkov 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **537** 022023

View the [article online](#) for updates and enhancements.



IOP | ebooks™

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

Electronic and microscopic researches of dislocation structure of metal near crack top

O A Nasibullina¹ and A S Tyusenkov

Ufa State Petroleum Technological University, Ufa 450062, Russian Federation

¹ E-mail: ksu33@bk.ru

Abstract. The most important element of fuel and energy complex is the system of the main pipelines for transport of gas, oil and products of their processing. On a surface of the main pipes in the presence of cathode protection there can be corrosion cracks. It is connected with simultaneous influence of two factors - soil electrolytes and the stretching tension. One of the most dangerous types of destruction of main gas pipelines is stress-crack corrosion (SCC). Defects on an outer surface of a pipe are shown in the form of single cracks or their system focused generally along the forming pipe. In work studying of the multiple cracks formed on the main gas pipeline under the exfoliated isolation is presented. Researches were conducted on a sample of X70 steel of the Japanese production which is selected from a focal zone of destruction of the main gas pipeline. On a sample there were cracks, characteristic of corrosion cracking. For the proof that cracks had corrosion and mechanical origin electronic and microscopic researches of dislocation structure of metal near crack top were conducted by means of a method thin foils and also the diffraction pattern of the site with a crack is studied.

1. Introduction

On the main pipe in use there can be cracks of various origin [1-7]. Main gas pipelines with the operating pressure of 5,5 - 10 MPas and with a diameter of 1020 - 1420 mm are the only metal designs in which fragile, very extended destructions were repeatedly observed [1, 2, 10-14]. The cracks having corrosion and mechanical origin, arising in the presence of cathode protection in places of peeling of insulating covering are the most dangerous. Corrosion cracking on straight-line-seam also the spiral-seam pipes, at the same time the crack which arose on the stress corrosion cracking mechanism energized takes place, itself is the concentrator of tension and at influence of static or cyclic loads becomes the initiator of destruction of a pipe. Defects on an outer surface are shown in the form of the single cracks or their system focused along the forming pipe

2. Experimental procedures

2.1. Materials

In work researches of X70 steel of the Japanese production were made. The sample was selected from a focal zone of destruction of the main gas pipeline on which there was a set of cracks, characteristic of corrosion cracking. Defects on an outer surface of a pipe were shown in the form of the systems of cracks, focused, generally along the forming pipe.



In figure 1 the studied sample of the main gas pipeline after preparation for researches - grindings and macroetchings in the mix of ice acetic and nitric acids (1:1) offered at MSCP USPTU department is presented.



Figure 1. Group of cracks on the surface of the tested sample.

Apparently in figure 1 the delay of distribution of the deepest crack when crossing the segregation strip having the hardness, big on 60 MPas, is observed than the average hardness of metal. Such behavior, apparently, is connected with influence of mechanical loads of metal at the time of accident.

2.2. Experiment Techniques

The first stage of all metalgraphic researches is production of samples (polished) which can be used for a microscopic research by means of light and electronic microscopes. Production of metalgraphic polished usually consists of five main operations: cuttings of a sample, its fixing (optional), grindings, polishings and etchings [5-9].

Cracks on the studied operating main gas pipeline arose on an outer surface of the pipeline within 5 - 7 hours of the conditional dial. It is revealed that development of refusal happened by formation of the main cracks; due to merge of their groups in the destruction center; and also due to formation of fistulas at through defeat of a wall of a pipe. At the same time the nature of development of destruction – fragile (from an outer surface of a pipe at an angle about 90 °) and with a viscous mechanical rupture area (at an angle about 45 °). In figure 2 disclosure of colony of cracks in the destruction center is shown.



Figure 2. Disclosure of colony of cracks at destruction of the pipeline (X70 steel).

Electronic and microscopic researches of dislocation structure of metal were conducted by means of a method thin фольг on an electronic microscope of JEM-2000 (accelerating voltage of 160 kV).

Linear defects are small in two measurements, in the third they can reach length of a crystal (grain). Dislocations are a special type of imperfection in a crystal lattice. From a position of the theory of dislocations durability, phase and structural transformations are considered. Dislocation is called the linear imperfection forming a shift zone in a crystal.

Thin a foil received by an electropolishing method. Electropolishing is the process of anode dissolution of metals which is followed by alignment of their surface. The reactants used for electropolishing can be rather poorly reactive. The electropolishing mode, electrolyte and also anode and cathode processes are rather fully characterized by a volt-ampere curve in coordinates "current

density – tension" on terminals of an electrolytic cell. If to stabilize anode potential potentiostat, having an additional electrode in a zone of possible formation of an opening, then the problem of production good foils significantly becomes simpler. Besides, thanks to a potentiostat it is possible to lower requirements to initial purity of a surface of a sample and to phase uniformity of material of a sample.

In work the diffraction pattern of the site of a sample of steel of X70 was submitted. Diffraction of electrons in an electronic microscope was reached following in the way - condenser lenses collimate a bunch, and all other lenses are switched off. When passing through a sample of almost parallel bunch of electrons there is a diffraction, and the picture is observed on the screen.

3. The results of studies and their discussion

By means of a method thin foils electronic and microscopic researches of dislocation structure of metal - near sample crack top X70 steel were made, further researches were conducted on an electronic microscope of JEM-2000 (the accelerating voltage of 160 kV). The sample was made by an electropolishing method.

Dislocations along with other defects participate in phase transformations, recrystallization, serve as the ready centers at loss of the second phase from solid solution. Along dislocations the speed of diffusion is several orders higher, than through a crystal lattice free of defects. Dislocations serve as concentration method of impurity atoms, in particular introduction impurity as it reduces distortion of a lattice. Impurity atoms form around dislocation a zone of the increased concentration which hinders the movement of dislocations and promotes metal hardenings. The listed defects of the crystal building lead to emergence of internal tension.

Results of researches are presented in figure 3.



Figure 3. A congestion of dislocations at crack top ($\times 30000$).

In figure 3 existence of tangle of dislocations near top of the second crack is observed that testifies to the high size of plastic deformation. This phenomenon is characteristic of corrosion and mechanical destruction.

Diffraction of electrons is extremely important source of information on structure of a sample. It is applied at researches thin metal foils and extraction remarks when the studied phase is taken from metal, or when studying the particles which are on a film substrate.

Diffraction picture gives an undistorted return crystal lattice of a solid body. However, the accuracy of determination of its parameters of a lattice is lower, than when using x-ray methods. Therefore, the electron diffraction is used, generally for determination of parameters of a crystal lattice of inclusions and orientation of crystals. The diffraction pattern of the explored site is provided on figure 4.

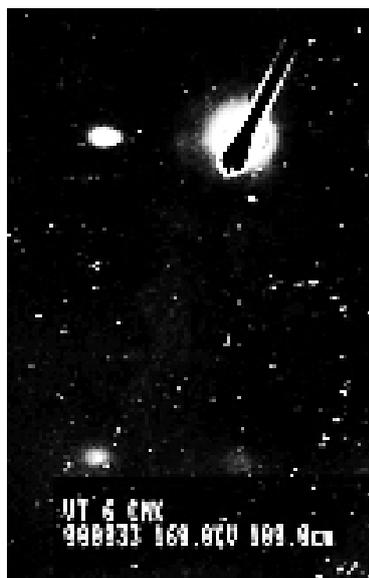


Figure 4. Electron diffraction pattern of the site.

Apparently from the provided photo in figure 4 diffraction reflexes correspond to a face-centered cubic lattice, the inverse volume-centered crystallattice α -Fe, lying in plane 111.

4. Conclusions

Electronic and microscopic researches of dislocation structure of metal near top of a crack revealed the tangle of dislocations near crack top demonstrating to the high size of plastic deformation that is characteristic of corrosion and mechanical destruction.

The diffraction picture gives an undistorted return crystal lattice of a solid body. Diffraction reflexes correspond to a face-centered cubic lattice, the inverse volume-centered crystallattice α -Fe, lying in plane 111.

Acknowledgments

The authors are grateful for the advice and assistance in conducting experiments Professor of the Ufa State Petroleum Technological University (Russian Federation) Gareev Alexei Gabdullovich.

References

- [1] Nasibullina OA, Gareev A G and Rizvanov R G 2018 *Solid State Phenomena* **284** 1302-6
- [2] Gareev A G, Rizvanov R G and Nasibullina O A 2016 *Corrosion and Protection of Metals in the Oil and Gas Industry* (Ufa: Gilem)
- [3] Nasibullina O A and Gareev A G 2019 *Materials Science Forum* **946** 381-6
- [4] Abdullin I G and Gareev A G 1994 *Materials Science* **29** (5) 539-41
- [5] Abdullin I G and Gareev A G 1993 *Fiziko-Khimicheskaya Mekhanika Materialov* **29** (5) 97-8
- [6] Tyusenkov A S 2016 *Steel* **2** 53-57
- [7] Tyusenkov A S, Rubtsov A V and Tlyasheva R R 2017 *Solid State Phenomena* **265** 868-72
- [8] Mirkhaydarova K A, Tyusenkov A S and Rizvanov R G 2018 *Solid State Phenomena* **284** 1297-301
- [9] Rizvanov R G, Abdeev R G, Matveev N L, Ryskulov R G and Shenknekht A F 2000 *Chemical and Petroleum Engineering* **36** 3
- [10] Kuzeev I R, Ibragimov I G, Bayazitov M I, Davydov S N and Khairudinov I R 1985 *Chem. and Technology of Fuels and Oils* **22** (3) 111-3
- [11] Ibragimov I G and Vil'danov R G 2007 *Welding International* **21** (2) 139-41
- [12] Rizvanov R G, Mulikov D Sh, Karetnikov D V, Fairushin A M and Tokarev A S 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **317** 012077

- [13] Tyusenkov A S and Cherepashkin S E 2014 *Russian Journal of Applied Chemistry* **87** (9) 1240-5
- [14] Karetnikov D V, Rizvanov R G, Fairushin A M and Kolokhov K S 2013 *Welding International* **27** (7) 557-60