Phase σ: Tracking down material defects

Phase σ: Tracking down material defects

Wanted: steel to handle extreme demands of petroleum/natural gas extraction

The quest for and extraction of petroleum and natural gas requires superior competence from both humans and materials involved. The demands made on both go far beyond normal conditions. The materials used must be produced using the best raw materials and produced at premium quality levels. Then even the tiniest fluctuations in the composition of the steel used for drills, drill rods and their casing can influence and decisively alter their required properties - such as degree of hardness, ductility and malleability.

Artem Yurovskikh, Ural State Technical University in Yekaterinburg, Russia, Dr. Manfred Kässens, Olympus Soft Imaging Solutions GmbH

This makes it no surprise that enormous efforts are being undertaken in the materials research area to a) better understand details of the chemical composition to be able to consistently obtain the quality desired, and b) to develop new, improved materials. At the Ural State Technical University in Yekaterinburg, Russia, Artem Yurovskikh and his colleagues are at work on these issues. Their current research project is investigating σ phases in Fe-28,5Cr-29,5Ni-3Mo-1,5Mn alloys. These secondary precipitation phases substantially influence the properties of the steels produced.

Energy - a best-selling export

In the course of the past few years, Russia has grown to be the second-largest energy producer in the world following the United States. 10 percent of primary energy worldwide originates in Russia. Russian oil reserves are estimated at a minimum of 50 billion barrels. That corresponds to a worldwide proportion of 15.6 percent of global reserves. This makes Russia the second-largest crude oil producer after Saudi Arabia. The situation is similar with natural gas. The largest natural gas reserves are on territory of what was once the USSR. Its successor states possess about 40 percent of these reserves. Russia has the largest portion of all.

Special materials for extreme conditions

Russian petroleum and natural gas reserves are primarily in Siberia. 70 percent of Russian petroleum and most of the Russian natural gas reserves are located there. The climate in Siberia is extremely harsh, which not insubstantially impairs and affects the extraction process. Permafrost is what makes walking on the ground or even erecting any construction possible. If the ground were to thaw, this would have dramatic consequences. This means that all buildings radiating any warmth have to be elabora-

1

Figure 1:
Artem Yurovskikh and his colleagues Anna Bannikova research phase and structural transitions in high-alloyed iron-chromium-nickel (Fe-Cr-Ni) alloy systems.

Figure 2:
The research of the precipitations and their origins requires a transmission electron microscope (TEM) like the JEOL JEM-2100 being used. This TEM is equipped with the Cantega G2 by Olympus Soft Imaging Solutions (OSIS), a 2k x 2k TEM camera. For image acqusition and image analysis Artem Yurovskikh and colleagues use iTEM, the OSIS TEM imaging platform and iTEM Solution Diffraction.

tely insulated. Without such precautionary measures they would sink into the soft boggy ground. The materials of the plants themselves must be up to handling the extreme outdoor conditions as well. The drills, the drill rods and their casings in particular are exposed to extreme heat, pressure and aggressive chemical conditions. This explains the intensive research being conducted on improving the equipment and developing innovative materials optimized for dealing with this uniquely extreme environment and the extraordinary demands involved in working in it. The Ural State Technical University in Yekaterinburg, Russia has a department called "Heat Treatment and Physics of Metal". Artem Yurovskikh and his colleagues Anna Bannikova and Sergey Belikov research phase and structural transitions in high-alloyed iron-chromium-nickel (Fe-Cr-Ni) alloy systems. These steels are some of the materials which are primarily used for producing drills and drill rod casings for the oil and gas extraction industry.

Intense demands on widely varied properties

The high-strength steels utilized in the oil and gas extraction in many Russian repositories require outstanding corrosion resistance despite extreme pressure. "What makes things even more challenging is that the ambient gases often contain H_2S-CO_2-Cl. These gases are very aggressive and attack the steel very quickly. The steel then corrodes", explains Yurovskikh. What researchers are looking for are high-grade steels which can withstand H_2S-CO_2-Cl-atmospheres as well. Chromium-nickel steels possess these properties and are superb for a whole range of industrial applications. "When sufficient nickel is used in corrosion-resistant steel", explains Yurovskikh, "then single-phase austentitic structures form. These structures are primarily responsible for the particular properties of these steels". This explains their widespread usage among the high-grade steels deployed in this context. Among other things, these particular properties result in a longer operational life span and enhance processing (of the steel).

Steels with diverse material properties

The material properties of the drills, drill rod casings and pump compressor pipes require a wide variety of characteristics. They need to insulate well, be resistant to stress and corrosion, but also be highly malleable and ductile for facilitating processing. Alloys with 25-30% chromium, ca. 30% nickel and 3% molybdenum offer these properties. Typical examples of these kinds of steel are Sanicro 28, Sm 2535 and DMV928. According to Artem Yurovskikh, an optimal structure for corrosion resistance is characterized "by a highly stable, single-phase, alloyed austenite". This austenite should consist of sufficiently large and homogeneous grains. In addition, continued Yurovskikh, the density of the crystalline defects in the material must be minimized and a low degree of residual stress is acceptable.

σ phases – undesirable precipitation

Current research at the Ural State Technical University in Yekaterinburg is focused on a special alloy: Fe-28,5Cr-29,5Ni-3Mo-1,5Mn. The mechanical characteristics of this alloy are as yet insufficient as demonstrated by its low impact toughness and limited yield stress. Preliminary investigation using light and scanning elec-

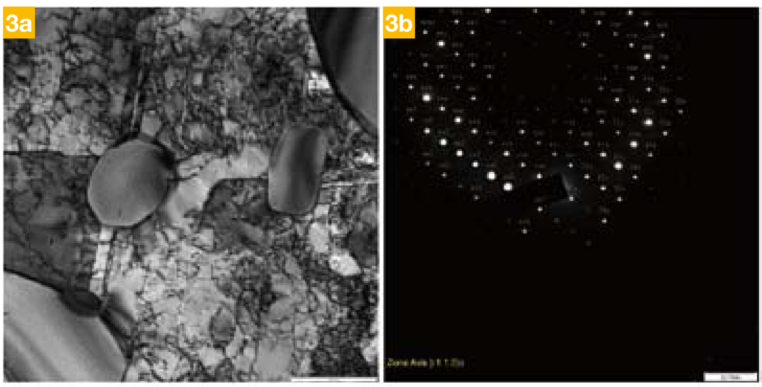

Figure 3:
Austenite with σ-FeCr precipitations in Fe-28,5Cr-29,5Ni-3Mo-1,5Mn alloy (a); SAD pattern corresponding to σ-phase (b)

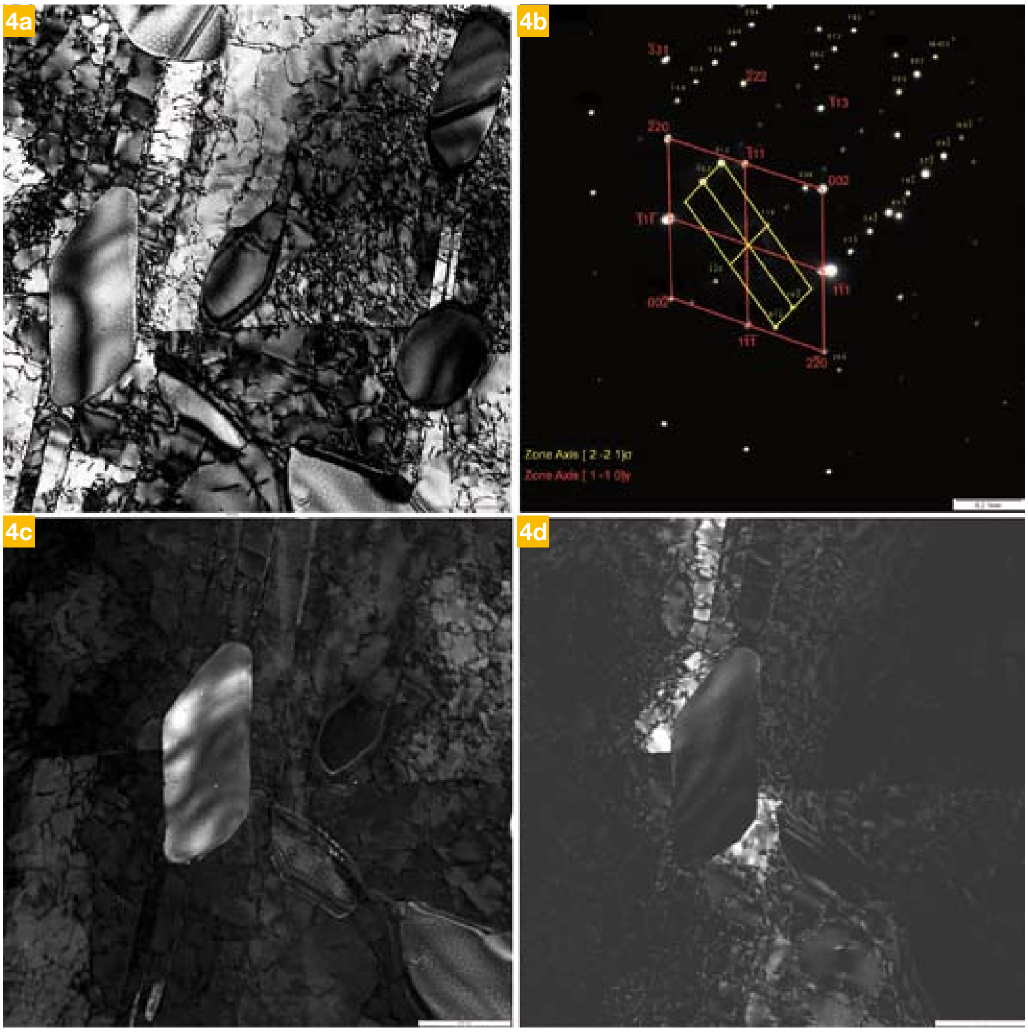

Figure 4:
σ-phase precipitation in austenite (a) and corresponding SAD pattern (b); dark-field image in (0-1-2) σ reflection (c); dark-field image in (1-11) γ reflection (d).

tron microscope methods indicate that a second precipitation phase in the alloy is a significant factor behind this.

The miniscule size of this precipitation means that detailed investigation is not feasible via light microscope (Olympus GX-51) or scanning electron microscope (JEOL JSM-6490). Further analysis and research of these precipitations and their origins requires a transmission electron microscope (TEM) like the JEOL JEM-2100 being used. This TEM is equipped with the Cantega G2 by Olympus Soft Imaging Solutions (OSIS), a 2k x 2k TEM camera. The image acquisition and subsequent evaluation of images is done using iTEM, the OSIS TEM imaging platform and iTEM Solution Diffraction, a special iTEM software extension. Investigation has shown that alongside large primary precipitation there are also smaller precipitations of a second phase. This second phase is primarily to be found along austenitic grain boundaries. This precipitation differs considerably from the surrounding matrix as its structure is practically dislocation free. More advanced evaluation and analysis via SAED (Selected-Area Electron Diffraction) - ie, diffraction investigation in selected areas - showed precipitates of a σ-similar FeCr phase corresponding to a primitive crystal tetragonal matrix.

“It is generally understood that the occurrence of σ phases in steels leads to a high degree of hardness combined with extremely low ductility and malleability as well as reduced fracture resistance”, explains Yurovskikh. “The presence of such a precipitate also lowers an alloy’s corrosion stability”, adds Yurovskikh. Chromium impoverishment when in the proximity of this precipitate is the reason. But it is not simply the decreased corrosion stability concerning the scientists, rather the alloy’s altered mechanical properties are the problem. “These can lead to crack formation during the tube shell cold drawing”, continues Yurovskikh. And this would seriously impair the entire production process.

Room for improvement

These results called for a determined response. Steps were taken to avoid the formation of σ phases during production. In order to optimize the steel’s chemical composition, a series of samples subjected to various heat treatment regimes has been explored. It became evident that σ phases can be suppressed within an acceptable grade composition of the Fe-28,5Cr-29,5Ni-3Mo-1,5Mn alloy. This was done by increasing the amount of nickel while at the same time reducing total chromium and molybdenum content. Furthermore, this alloy's composition requires meticulous observance of time-temperature parameters of tube & shell heat treatment – during all steps of the production process. Special attention should be paid to the round billet cooling speed after piercing, because low cooling speed promotes the formation of σ phases. Following the proposed recommendations has facilitated production of the necessary uniphase austenitic structure in Fe-28,5Cr-29,5Ni-3Mo-1,5Mn alloys – free of the second phase precipitations.

Conclusion

Extracting petroleum and natural gas is a tremendous challenge for the people and materials involved. This is why materials are needed which can stand the extreme outdoor conditions as well as withstands extreme material wear and tears. Artem Yurovskikh and the team at the Ural State Technical University in Yekaterinburg specializes in investigating properties of high-grade steels such as the Fe-28,5Cr-29,5Ni-3Mo-1,5Mn alloys. Due to their specific parameters these are the steels primarily employed in petroleum and natural gas extraction. Yurovskikh's work has obtained critical information on avoiding formation of σ phases in this alloy. These precipitates are a significant impairment to production of drills, drill rods and casings.

ACKNOWLEDGE:

Text written in close collaboration with:
Artem Yurovskikh, Anna Bannikova, Sergey Belikov,
Heat Treatment and Physics of Metals department,
Ural State Technical University, Ekaterinburg, Russia

Image source:
Artem Yurovskikh, Ural State Technical University in
Yekaterinburg, Russia
Kevin Abbott, USA, Title image

Application Report 4-931-201-09-1,02-2009

www.olympus-sis.com
www.soft-imaging.net

OLYMPUS

OLYMPUS SOFT IMAGING SOLUTIONS GMBH
Johann-Krane-Weg 39
48149 Münster
Germany
Phone: +49 (251) 7 98 00-0
Fax: +49 (251) 7 98 00-99
info.osis@olympus-sis.com

OLYMPUS SOFT IMAGING SOLUTIONS CORP.
200 Union Blvd, Suite 202
Lakewood, CO 80228
USA
Phone: +1 (303) 234-9270
Fax: +1 (303) 234-9271
info.osis@olympus-sis.com

OLYMPUS SOFT IMAGING SOLUTIONS PTE.LTD.
No 2 Jurong East St 21 #05-30
IMM Building, Singapore 609601
Singapore
Phone: +65 (6777) 7898
Fax: +65 (6777) 7218
info.osis@olympus-sis.com