Friction Induced Structural Changes of Mangalloy Observed by Synchrotron X-Ray Diffraction and Metallographic Analysis

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Abstract. The effect of friction impact on structure and properties of mangalloy (Hadfield steel) was investigated. By means of metallographic analysis coupled with synchrotron X-ray diffraction in operando regime, the microstructural changes in specimen's subsurface layer during pin-on-disk experiment were studied. Analysis of phase composition showed that formation of martensite side by side with austenite has a cyclic behavior. Besides, it was shown that overall hardening is associated with a set of structural transformations. Finally, the failure of surface layer was observed.

1. Introduction
To date a lot of different steel grades for mold casting are known. The high-manganese steels that are one of the most common among iron-carbon alloys designed for operation under high contact loads of particular importance [1]. The development of mangalloy by R. Hadfield over 100 years ago was a starting point for its investigation. The chemical composition of Hadfield steel is shown in table 1. Known, that such alloys are characterized by high wear resistance at high specific and impact loads as well as by high degree of plasticity. Meanwhile, application of high manganese steels is limited due to extremely poor machining, metal forming and relatively low initial hardness. These alloys are mainly used for cast parts that do not undergo subsequent machining [2].

| C       | Mn         | Si         | Cr | Ni | Cu | S   | P   | Fe   |
|---------|------------|------------|----|----|----|-----|-----|------|
| 0.90 - 1.40 | 11.50 - 15.00 | 0.30 - 1.00 | 1.0 | 1.0 | 0.30 | 0.050 | 0.120 | balance |

It's a well-known fact that high strength of austenitic steels is usually achieved by cold working instead of heat treatment. In doing so, one of strengthening mechanisms is a strain induced formation of α'- and ε-martensite as it was demonstrated in the first half of the 20th century [2]. Though, high degree of work hardening was also observed in the absence of these structural components that was confirmed by X-ray diffraction analysis. The mechanical twinning was found in such cases [2-5] and most of the studies were conducted using light and electronic (both scanning and transmission) microscopy.
Due to the wide spread of high manganese iron carbon alloys like Hadfield steel the mechanisms of their strengthening still require detailed consideration. The microstructural evolution under sliding friction are of special interest since it could combine cyclic loading, severe plastic deformation and heating. The non-destructive methods based on microscopy are typically used to study features of structural transformations. However, this approach is not always rational since an experimental sample subjected to certain number of friction cycles cannot be subjected to further tests after microscopic studies. Therefore, a new sample is required to study subsequent structural alterations caused by higher number of revolutions. This procedure, in turn, can lead to a mismatch in the test conditions. In addition, the interruption of the tribological process can lead to the removal of wear debris, cooling or heating of the studied object during sample preparation for metallographic observation that leads to a change in the structure of the subsurface layers, the thickness of which is often units of micrometers. It is for that reason that methods of structural evolution characterization using synchrotron radiation is of particular interest [6]. High brilliance of radiation provides an opportunity to obtain hundreds of diffraction patterns per second in operando or in situ regimes. This paper presents the results of the dry sliding friction process of Hadfield steel obtained with friction tester with simultaneous use of synchrotron microdiffraction.

2. Experimental procedure

2.1. Description of the experimental scheme

The test was carried out according to the «pin-on-disc» friction geometry at the ID13 beamline of the European Synchrotron Radiation Facility (ESRF). A schematic representation of the experiment is shown in figure 1. The beam energy and the beam size were 13.9 keV and 2.8×1.3 μm respectively. The inclined radial surface of the disk was irradiated using synchrotron beam behind the pin at a distance of approximately 3 mm. The slope of 3.4° degrees contributed to an increase in the beam projection on the studied surface up to 2.8×22 μm. During friction diffracted beam was recorded using two-dimensional detector DECTRIS EIGER 4M with resolution 2070×2167 pixels and a maximum frequency 750 Hz. Diffraction ring patterns have been azimuthally integrated and analyzed using scripts developed in the Python programming language. Instrumental broadening was taken into account via α-Al2O3 standard. A detailed description of the experiment is presented in previous works [7, 8].

![Figure 1. A scheme of a pin-on-disk experiment with simultaneous synchrotron X-ray diffraction: (a) – incident synchrotron beam; (b) – diffracted beam; (c) – 2D detector; (d) – disk-shape sample; (e) – WC-Co pin; (f) – trace of the incident beam; (g) – sliding direction.](image)

The disc sample was made of high manganese austenitic Hadfield steel (elemental composition is shown in Table 2) via lathe machining. The radial surface of the disc was carefully polished using
diamond pastes after which vacuum quenching was carried out with a temperature of 1000 °C in the oil. No traces of oxidation were observed on the surface of the sample after heat treatment. It was assumed that the sample preparation eliminated all the effects of previous machining and that any remaining structural imperfections, such as microstresses, dislocations, etc. were only a consequence of the heat treatment.

Table 2. Elemental composition of studied material.

|   | C    | Mn   | Si   | Cr | Ni  | Cu | S  | P  | Fe |
|---|------|------|------|----|-----|----|----|----|----|
|   | 1.060| 12.750| 0.79  | 0.070| 0.062| 0.255 | 0.005 | 0.045 | balance |

A pin was made from tungsten carbide-cobalt alloy. This material was chosen because of its high mechanical properties, in particular high wear resistance. During the experiment, the steel sample was pressed against a rigidly fixed pin. This approach allowed to keep the position of the disk surface unchanged when it wears out.

The experiment was implemented under dry friction conditions, the contact pressure was 160 MPa, and the angular velocity was 10 rpm.

2.2. Methods of material characterization

Upon the completion of the experiment the sample was cut using a wire electrical discharge machining along the direction of friction and perpendicular to the axis of rotation. Surface preparation was carried out by grinding on abrasive paper and polishing using diamond suspensions. The microstructure was investigated with scanning electron microscope (SEM) Carl Zeiss EVO50 after etching in a solution of nitric acid and water in a ratio of 1:9. Microhardness was measured by the Vickers method at a load of 0.098 N in three regions at different distances from the contact surface, each value was obtained by averaging 5 measurements. The elemental composition of the alloy was analyzed using optical emission spectrometer ARL 3460.

3. Results and discussion

3.1. The evolution of phase composition

The studies conducted using synchrotron X-ray diffraction have shown that structure of subsurface layers consist of austenite and martensite mixture until at least 100 friction cycles (figure 2). The presence of the ε-phase was observed only once after 30 cycles of friction.

Figure 2. X-ray diffraction patterns obtained at different stages of friction with the use of synchrotron radiation.
An unexpected result is the presence of $\alpha'$-martensite at the beginning of the experiment. Probably, its presence is due to the deformation of the material during quenching that could be caused by thermal stresses associated with the high cooling rate.

It may be noted that the alteration in the height of martensite peaks in dependence on number of revolutions has a cyclic behavior. It is also aware that the relative intensity of a single peak of a certain phase is associated with the atomic structure of the crystal and the volume fraction of this phase as well as significantly depends on the presence of crystallographic texture [9]. In addition, alteration of peak shape in particular the full width at half maximum (FWHM) is associated with subgrain refinement, lattice distortions and accumulation or annihilation of dislocations [10]. Based on the analysis of the obtained results including figure 3 we believe that the accumulation of damage of the subsurface layers that lead to the strain induced transformation of austenite into martensite, its subsequent deformation, failure and removal in the form of wear debris are observed from the outset. At the same time, «fresh» - lightly deformed areas of $\gamma$-iron are opened then the described process is repeated. So, for instance, upon ten-twenty friction cycles the relative intensity of supersaturated solution $\alpha'$ decreases compared to the diffraction maximum of austenite. Apparently, it is associated with fracture and removal of $\alpha'$-martensite from the contact area and the appearance of fresh $\gamma$-phase regions on the sample surface.

Further, the austenitic structure adapts to external loads that is expressed in dislocation glide as well as the rotation of the microvolumes of the specimen in which slip planes are oriented parallel to the sliding direction [11]. In the meantime, we can observe the increase in austenite’ FWHM. And then after the 50-th revolution the fraction of austenite increases again to which indicates repeating the of the above described process.

![Figure 3. Diffraction line fitting (a) and alteration of FWHM in dependence on friction cycles (b).](image)

3.2. The fracture of subsurface layer

Figure 4 shows the surface of a high manganese steel disc subjected to friction loading. The structure of the contact spot allows to conclude that severe plastic deformations occurs on the surface which contributed to the separation of the material and formation of wear debris involved in friction interaction [12]. Besides, it is possible to note the presence of microcracks, which also confirm the assumption of the destruction of the surface layer, leading to the removal of martensite from the contact region [11, 13].
The microstructure pictures obtained from the longitudinal section of the sample show that microcracks propagate in direction perpendicular to the friction surface (figure 5). This behavior is typical for delamination processes. The thickness of the surface layer, presumably consisting of a mixture of austenite, martensite, and wear products is approximately 1 µm. The area below is austenite grains with densely spaced slip bands. The distribution of microhardness from the contact surface at a distance from the surface of 10, 70 and 205 µm is 576.0±106.8, 458.2±45.2 and 396.0±48.8 HV, respectively. Thus, strengthening is associated with grain refinement, slip bands and dislocations as well as precipitation of strain induced martensite which probably has nanoscale size. Nevertheless, the hardening mechanism requires more detailed consideration, including the study of the fine structure of the material.

4. Conclusions
Based on this study, it can be concluded that the hardening of the deformed layer is achieved with a set of structural transformations and not limited to strain induced α’-martensite generation. Formation of slip bands, structure refinement and rotation of microvolumes also contribute to increasing of hardness. It is shown that the presence of martensite in the interaction region has a cyclic behavior due to the processes of deformation-induced transformation and delamination.

Acknowledgments
The reported study was funded by RFBR and Novosibirsk region according to the research project № 19-48-543022.
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