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EBSD STUDY OF CORROSION FATIGUE OF AUSTENITIC-FERRITIC STEEL[1)]

BADANIA KOROZJI ZMĘCZENIOWEJ W STALI FERRYTYCZNO-AUSTENITYCZNEJ METODĄ EBSD

Fatigue crack propagation investigations have been performed in austenitic-ferritic duplex stainless steel 00H22N5M3 in air and during hydrogen charging, using various frequencies of loading. Strong differences of crack propagation velocity depending on the test conditions were noticed. Lower frequency with applied hydrogen charging led to the huge increase of crack propagation velocity compared to the tests performed in air. To understand such a behaviour in each case and characterize crack mode, the samples were observed using electron back-scattered diffraction (EBSD). It was shown that in air, the fatigue crack propagation involved plastic deformation and the resulting cracks had ductile character. The presence of hydrogen led to more brittle mode of cracking. This effect was also connected with frequency of loading: lower frequency, which assured longer time for hydrogen-crack tip interaction, resulted in the highest crack propagation velocity and the brittle cracking mode with lower amount of plastic deformation. The performed observations indicated that the path of the crack went mostly transgranularly through both austenite and ferrite phases. Phase and grain boundaries were not the preferred paths for crack propagation.

Keywords: EBSD, duplex steel, hydrogen, SEM

Tematem pracy były badania propagacji pęknięć zmęczeniowych w niskowęglowej dwufazowej stali ferrytyczno-austenitycznej typu 00H22N5M3 w atmosferze otoczenia oraz w warunkach nasycenia wodorem. Stwierdzono występowanie istotnych różnic w prędkości rozprzestrzeniania się pęknięć w zależności od rodzaju testów. Niższe częstotliwości cyklicznego obciążenia w połączeniu z ładowaniem wodorem prowadziły do znaczącego wzrostu prędkości propagacji pęknięć zmęczeniowych w porównaniu do testów przeprowadzonych w powietrzu. Celem prześledzenia przebiegu pęknięć zmęczeniowych poprzez materiał oraz określenia ich charakteru próbki były zanalizowane przy użyciu dyfrakcji elektronów wstecznie rozproszonych EBSD. Rozprzestrzenianie się pęknięć w powietrzu prowadziło do odkształceń plastycznych dając w rezultacie pęknięcia o charakterze ciągłym. Obecność wodoru prowadzi do bardziej kruchego pękania. Efekt jest również związany z częstotliwością cyklicznego obciążania. Niższe częstotliwości, połączone z dłuższym oddziaływaniem wodoru z koniuszkiem pęknięcia, prowadzą do maksymalnej prędkości propagacji. Pękanie ma charakter kruchy i praktycznie nie istnieje wokół niego obszar zdeformowany. Przeprowadzone obserwacje udowodniły, iż bieg pęknięcia ma charakter transgranularny zarówno w fazie austenitycznej jak i ferrytycznej. Propagacja pęknięć nie występuje wzdłuż granic międzyfazowych i międzyziarnowych.

1. Introduction

Austenitic-ferritic duplex stainless steels are modern materials characterized by very good mechanical properties with good corrosion resistance. Therefore, they find many applications in the petro-chemical and offshore structure industries. However, their successful exploitation can be limited by the hydrogen embrittlement phenomenon, which can take place in certain conditions (for example use of cathodic polarisation). Hydrogen presence provokes distinct reduction of plastic properties, what was shown in tensile test experiments [1, 2]. The performed investigations indicate that one of the most important conditions for hydrogen embrittlement is plastic deformation [3]. Materials used in the industrial structures are rarely exploited at loads higher than the yield point, but local stresses may exceed this value in the areas where the stress concentration appears:

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structural or constructional notches. This process may be additionally intensified by the repeating cyclic loading which leads, in the presence of hydrogen, to corrosion fatigue phenomenon. Therefore, the verification of susceptibility of duplex stainless steel to the hydrogen corrosion fatigue may be crucial for the practical application of this material.

In the literature, one can find very little information about this problem, mostly about the influence of some specific environments like seawater [4], gaseous hydrogen [5] or NaCl solution [6]. However, a lot of aspects still remain not clarified, for example the influence of the steel microstructure. Our earlier investigations [7], performed in the conditions of cathodic hydrogen charging, revealed changes of the crack propagation velocity compared to tests performed in air. The observed effect was stronger for lower frequency of loading (0.2 Hz against 20 Hz). Therefore, some questions arise: what was the mode of cracking and the path of the crack – was any of the phases more privileged for cracking? And were there any influences there of phase or grain boundaries on the cracking? In order to resolve these problems EBSD has been applied since orientation measurements along cracks should give at least a trend inducting answer to these questions.

2. Experimental

The material used in this study was Cr23-Ni5-Mo3 austenitic-ferritic duplex stainless steel in the form of the sheet of 6 mm of width (Fig. 1).

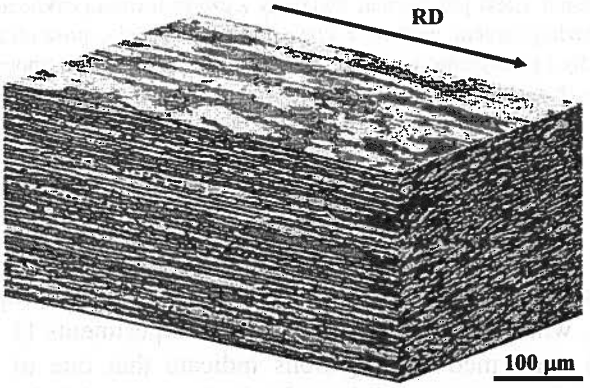

Fig. 1. Microstructure of Cr23-Ni5-Mo3 austenitic-ferritic duplex stainless steel

The fatigue crack propagation investigations have been performed in air and during hydrogen charging, using various frequencies of loading: 0.2 Hz and 20 Hz. Grinded and polished compact tension (CT) specimens were used for the crack growth test (Fig. 2). The crack growth occurred parallel to the rolling direction of the samples and perpendicular to the loading direction. Cathodic hydrogen charging was performed electrolytically from 0.1 M H_2SO_4 aqueous solution, with addition of hydrogen entry promoter, at room temperature.

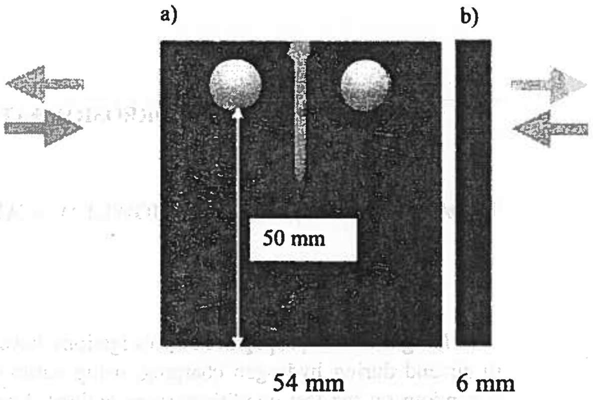

Fig. 2. Compact tension (CT) specimen used for the crack growth tests: a) front view, b) side view

Then, the samples were observed using the EBSD technique. The analyses were performed in a field emission gun scanning electron microscope (FEG-SEM) LEO Gemini 1530VP with an attached EBSD system of HKL Technology. The data post-processing was carried out using CHANNEL5 software.

3. Results and discussion

The EBSD phase discrimination in the steel samples enables characterization of the fatigue crack path through the material. It reveals the austenite and ferrite distribution (Fig. 3). Several layers of the material have been removed by polishing. Consecutive observations were performed in order to track the crack path inside the material. Finally, from numerous measurements it has been concluded that there is no phase more privileged for cracking. The path of the crack goes mostly transgranularly through both the austenite and ferrite grains. Even though the fatigue tests were performed in parallel to the rolling direction, i.e. strong texture can be observed, the material does not show any tendency to splitting along phase boundaries. It also seems clear that grain boundaries of the duplex steel are not the preferred paths for crack propagation.

The so-called band contrast (BC) maps allows us to discover the variation of cracking mode depending on the test conditions. It is possible, because the diffraction signals – the bands – lose their sharpness and contrast e.g. in regions with high dislocation density (short coherence length). Transformed in a grey scale these regions appear as darker areas in the BC map. The non-indexed crack zone (void) is also visible as dark area. In a first approximation one can assume that the variation in cracking mode between the samples appears as differences in the thickness of the dark zone around a crack in the BC map. The higher the dislocation density, i.e. the more ductile mode of cracking, the thicker is the non-indexed dark zone around a crack itself. Another possibility of displaying deformation in a material using EBSD orientation data are misorientation maps. The orientation deviations of all analysed points in relation to a reference orientation are used to reflect the existing lattice strain in a single grain. The band contrast analysis shows distinct differences between the samples tested in various conditions. The largest dark zone around the crack is displayed in the sample tested in air (Fig. 4a), whereas the narrowest is given for the sample tested in hydrogen with 0.2 Hz frequency (Fig. 4b). From this it can be concluded that for a sample without hydrogen charging, typically a ductile mode of cracking is observed. Hydrogen charging provokes the change of this cracking mode to more brittle. The level of brittleness depends on the loading frequency: a lower frequency assures longer time for hydrogen-crack tip interaction, and reduces the plastic deformation. It also results in higher crack propagation velocity.

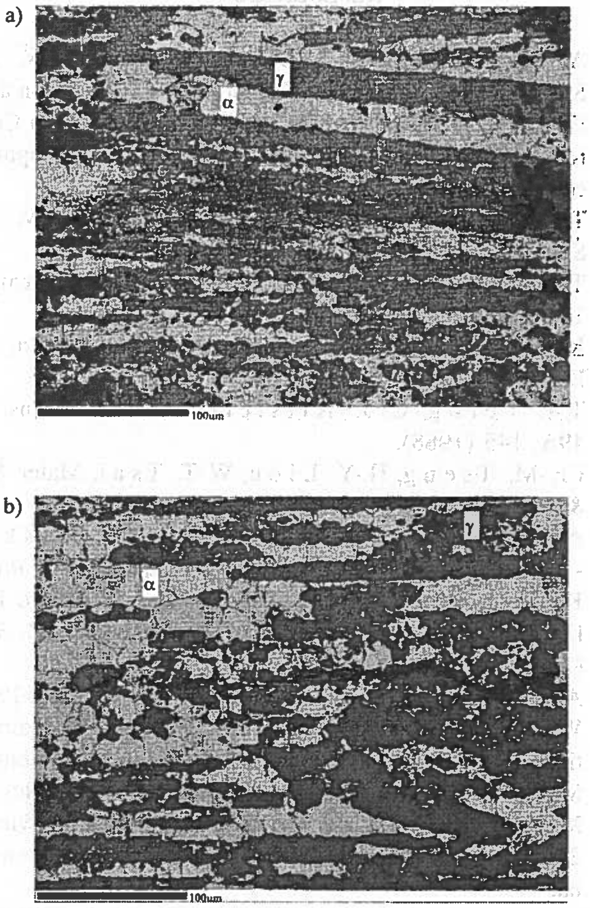

Fig. 3. Phase distribution in the steel: ferrite (bcc) – red, austenite (fcc) – blue; a) sample tested in air, b) sample tested in H_2SO_4 solution (0.2 Hz)

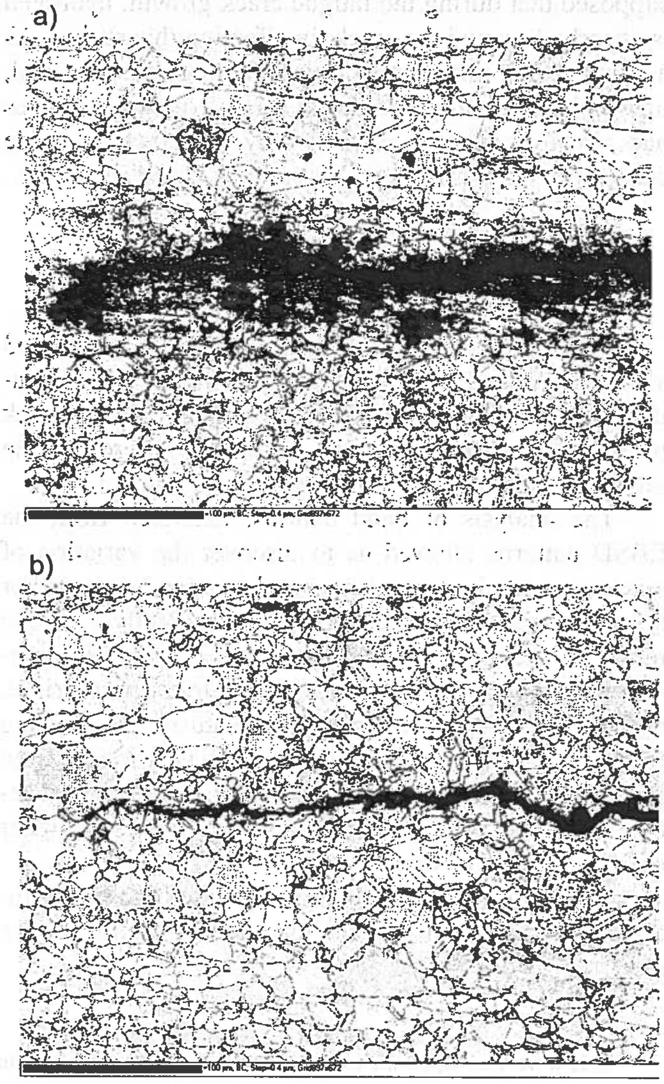

Fig. 4. Band contrast analysis: a) sample tested in air, b) sample tested in H_2SO_4 solution (0.2 Hz)

Stronger hydrogen effects observed in case of lower frequency of 0.2 Hz are directly connected with longer time of one fatigue cycle (5 s for 0.2 Hz against 0.05 s for 20 Hz). As a result, it enables a longer time for the crack tip interaction with hydrogen during one cycle. It can be supposed that hydrogen absorbed in the crack tip area interacts with the steel microstructure, changing its properties. The SEM and EBSD analyses do not reveal any phase transformation in this region. Martensitic transformation ($\gamma \rightarrow \alpha$') is observed only on the sample surface [8], but along the crack path martensite has not been detected.

Our earlier observations of hydrogenated samples using transmission electron microscopy [9] revealed that hydrogen absorption in steel resulted in the strong increase of dislocation density in ferrite and stacking faults in austenite. These microstructural changes take place only in a very thin surface layer. Therefore, it could be supposed that during the fatigue crack growth, hydrogen is absorbed around the crack tip affecting this region. As a result, crack growth takes place through the material, in which the hydrogen induced microstructural changes had already occurred. That is why the cracking mode changes in comparison to the samples tested in the air.

4. Conclusions

The EBSD analysis of Cr23-Ni5-Mo3 austenitic-ferritic duplex steel showed that both austenite and ferrite phases are equally susceptible to crack propagation. Phase and grain boundaries were not the preferred paths for crack propagation.

The analysis of band contrast extracted from the EBSD patterns allowed us to discover the variation of cracking mode in dependence on the test conditions. For a sample without hydrogen charging, typically a ductile mode of cracking was observed. Hydrogen charging provoked the change of this cracking mode to more brittle, and the degree of brittleness depended on the loading frequency. A lower frequency resulted in a longer time for hydrogen-crack tip interaction and therefore in a reduced ductile behaviour. Moreover, it causes a higher crack propagation velocity.

The performed investigations revealed the strong influence of hydrogen on cracking mode of the duplex steel.

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