Transactions of the VŠB – Technical University of Ostrava, Mechanical Series
No. 1, 2011, vol. LVII
article No. 1836

Vladimír CIHAL[*,*], Marie BLAHETOVÁ[*], Eva KALABISOVA[***], Zdena KRHUTOVÁ[*], Stanislav LASEK[*], Jiri MIKA[**], Libor TUREK[***]**

SENSITIZATION TO CORROSION AS INITIATOR OF FATIGUE FRACTURE IN COMPRESSOR BLADES
ZCITLIVĚNÍ KE KOROZI JAKO INICIÁTOR ÚNAVOVÉHO LOMU LOPATEK KOMPRESORU

Abstract

Certain failures of stainless steels interpreted purely in terms of fracture mechanisms may in fact be closely associated with previous damage caused by localized corrosion. The closeness of the link between fatigue and corrosion is documented by the case history of compressor blades made of grade 14Cr17Ni2 (X14CrNi17-2) stainless steel. Fatigue fracturing observed in areas near the blade root tended to follow intergranular pathways, indicating that some additional mechanism other than fatigue might be involved. This suspicion was confirmed by electrochemical potentiokinetic reactivation (EPR) measurements in situ, which revealed sensitization to intergranular corrosion. It has been found that at the transition between the blade root and the blade proper the surfaces had been ground and polished too vigorously, heating the subcutaneous layers to within the danger zone of 400-600°C. Preferential integranular attack in these locations was the initiation mechanism that provoked a subsequent failure of the blades by fatigue fracture.

Abstrakt

Určité poruchy korozivzdorných ocelí zdůvodňované čistě ve smyslu lomové mechaniky mohou být ve skutečnosti úzce spojeny s dřívějším poškozením způsobeným místní korozí. Souvislost mezi únavou a korozí představuje případ lopatek kompresoru vyrobených z korozivzdorné oceli typu 14Cr17Ni2. Únavové poškození (praskání) pozorované v oblastech blízko paty lopatky má tendenci sledovat mezikrystalovou cestu. To naznačuje, že přichází v úvahu i jiný mechanismus vzniku trhlin, než je únava materiálu. Toto podezření bylo potvrzeno elektrochemickým potenciokinetickým reaktivačním (EPR) měřením „in situ“, které odhalilo zcitlivění k mezikrystalové korozi. Bylo zjištěno, že v přechodu mezi patou lopatky a ostatními povrchy byla broušena a leštěna příliš razantně a došlo k ohřátí povrchových vrstev v rozsahu teplot 400-600°C. Přednostní mezikrystalové napadení v těchto místech bylo iniciačním mechanizmem pro vyvolání následujícího poškození lopatek únavovým lomem.

1 INTRODUCTION

The behavior of material in terms of fracture mechanics may in some cases be closely related to a damage suffered by the material due to corrosion. The closeness of this link can be documented by a case which occurred in practice: compressor blade made of grade 14Cr17Ni2 steel suffered localized corrosion attack near the blade foot in areas previously ground and polished.

The electrochemical polarization methods came as a response to a situation in corrosion testing which was primarily characterized by the need for a better, quantitative method of measuring

[*] Marie BLAHETOVÁ, Zdena KRHUTOVÁ, Stanislav LASEK VSB – Technical University, Faculty of Metallurgy and Materials Engineering, Department of Materials Engineering, 17. listopadu15/2172, 708 33 Ostrava

[***] Eva KALABISOVA, Libor TUREK,SVUOM Ltd., V Šáreckém údolí 2329, 164 00 Prague

[**] Jiri MIKA, VSB – Technical University, Faculty of Mechanical Engineering, Department of Energy Engineering, 17. listopadu15/2172, 708 33 Ostrava

the degree of sensitization (*e.g.*, in welded components) and the need for a rapid test that could also be applied in the field [1]. Most widespread of these became the potentiodynamic measurements where a scan is run across a range of linearly changing, controlled potentials of the working electrode (metal specimen) and the measured current response is an integral curve in the sense that the current density coordinate of the polarization plot corresponds to the algebraic sum of all oxidative and reductive processes taking place on the surface of the working electrode under study.

The EPR test came to be developed at a relatively early stage of electrochemical testing, as a special subgroup of the potentiodynamic measurements. Double-loop EPR, applied in the present case study, is a bidirectional, 'cyclic' sweep consisting of a forward scan followed by a reverse scan, possibly with a delay at vertex which generally is located within the passive range. Various quantities (mainly, characteristic potentials, currents and coulombic charges) are recorded and interpreted as relating to the different corrosion types studied, but they all derive from the shape of the EPR curves measured.

An early example of application of double-loop EPR is Fig. 1 demonstrating the original-historical-experimental conditions, developed and employed in 1969 by Cihal [2] in Centre de Recherches de Firminy C.A.F.L. in France to detect TiC carbides and Ti_xS sulfides. The method involved the use of H_2SO_4 at 70°C under N_2, and polarization scans at 9 V/h, also detecting reactivation due to chromium carbide grain boundary precipitation and intergranular sensitivity.

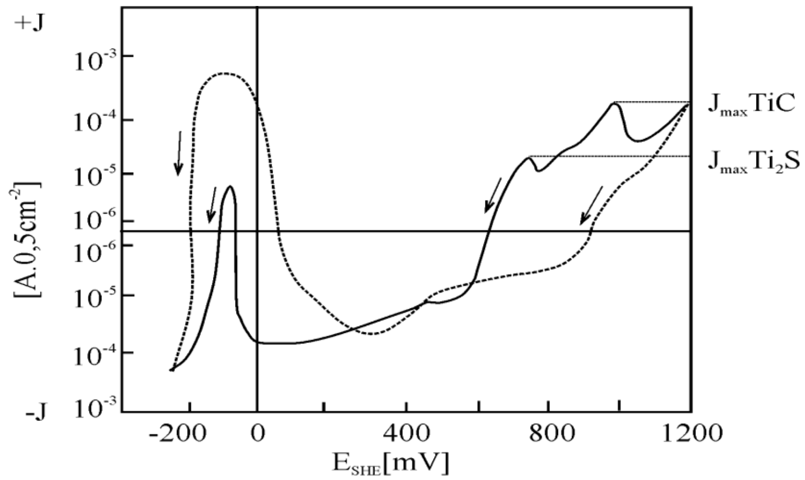

Fig. 1 Reverse scans of double loop polarization curves for annealed grade Cr18Ni12Ti steel in 2.5M H_2SO_4. —— 1150°C/20 min/water +590°C/20 h/water
------ 1300°C/20 min/water +550°C/20 h/water.

Since then, the EPR test, designed to examine the susceptibility to nonuniform, primarily intergranular corrosion, has come to rank among the more successful testing technique developments relating to stainless steels and alloys. One of its numerous advantages is that it lends itself to non-destructive, on-site examination. Prominent applications of the EPR technique include a variety of alloys and their phases in various corrosion environments [3].

2 CASE HISTORY: CORROSION-INITIATED FATIGUE FAILURE

The corrosion-and-fatigue failure case described below concerns the blades of a high-power air compressor (Fig. 2). The compressor suffered damage both on the rotor blades (Fig. 3) and on the stator blades. The fact that stator blades were also attacked indicated that fatigue could not have been the decisive factor contributing to corrosion. It appeared that, conversely, corrosion was a factor responsible for the onset of fatigue.

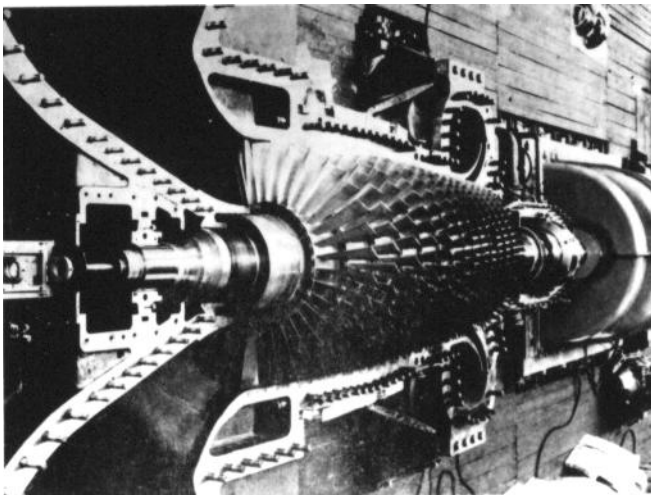

Fig. 2 View of the failed air compressor (exhaustor).

It was suspected from the outset that the corrosion damage observed had not been produced while the compressor was running but, to the contrary, during a period of compressor shutdown. The corrosion medium responsible was the condensate – produced by condensation from humid air. Certain areas of the blades, dismantled from the compressor after a period of operation, were found to have suffered corrosion damage of intergranular character. Also, fatigue tests performed on the blades indicated inadmissible, too low values of the fatigue limit in some cases [4]. The fatigue fracture surface showed certain amounts of intergranular facettes.

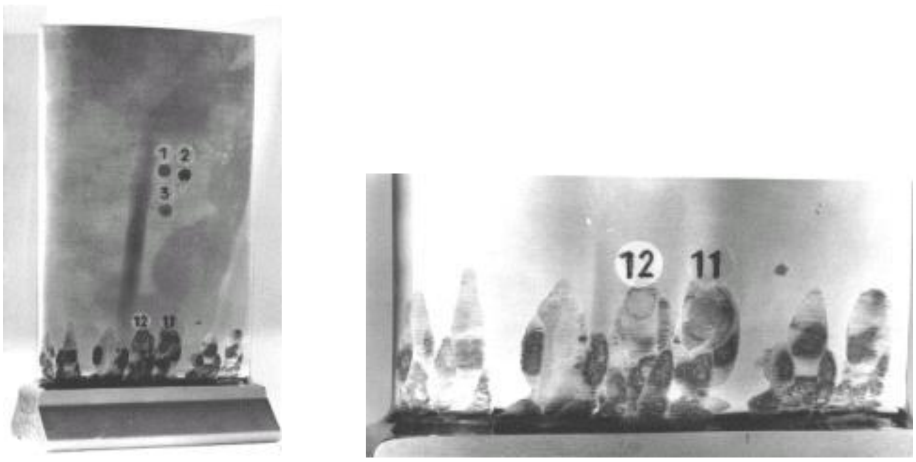

Fig. 3 Corrosion and fatigue damage on rotor blade indicating EPR test locations:
view of blade (left) and detailed view of blade base (right).

It had been presumed initially that the low fatigue limit might be related to some early-stage manufacturing operations where in certain cases, extensive cracks of markedly intergranular character were found, the material also exhibiting catastrophic local reductions of notch toughness. It was initially believed that the damage suffered at the points of transition between the blade proper and its base was due to a fatigue mechanism or that possibly, so-called corrosion fatigue was involved. Eventually however, the examination of the affected areas (also relying on previous experience from research of austenit-martensitic steels [5]) led us to suspect a different mechanism, *viz.*, that at the transition between the blade root and the blade the pressures applied during the grinding and polishing operations were excessive, provoking a primary sensitization to corrosion in the material where the temperatures reached the danger zone of 400-600°C, *i.e.*, that the material became corrosion-sensitive due to local overheating.

3 EXPERIMENTAL

The method chosen to examine the cause underlying this damage was EPR— electrochemical potential reactivation. The EPR technique was applied *in situ* to the locations identified in the Fig. 3.

The medium used for the EPR measurements was 0.5M H_2SO_4 + 0.01% KSCN, and the potentiodynamic scans were run both on the blades (mainly focusing on the "singed" areas) and on comparative specimens, at a potential scan rate of 250 mV/min. and a temperature of 25°C. The quantities recorded were the critical passivation current density J_p, the repassivation current density J_r, the coulombic charge passed during activation and passivation C_p, and the coulombic charge passed during repassivation and reactivation C_r.

The electrochemical measurements were confronted with conventional metallographic and SEM images.

4 RESULTS

Metallography for the heat-affected ("singed") zones of the blades suggests an increased sensitivity to intergranular corrosion [6] of the blades, due to the precipitation at grain boundaries of chromium carbides (Fig. 4).

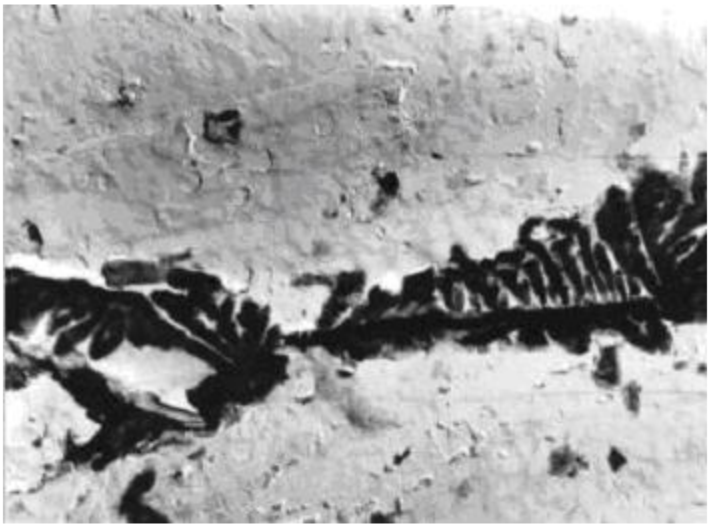

Fig. 4 Extraction thin foil with precipitated carbides (5000x).

The results of the potentiodynamic tests run on the heat-affected surfaces of the blades are given in Table 1. Two or three consecutive measurements were run at some places of the blade surface (in a "singed" region).

Table 1 EPR measurements on heat-affected surfaces of the blades.

test location			J_p $mA \cdot cm^{-2}$	J_r $mA \cdot cm^{-2}$	C_p $C \cdot cm^{-2}$	C_r $C\ cm^{-2}$	C_r/C_p [%]
rotor	base	11	22.0	7.9	0.972	0.280	28.8
		12	180.0 19.6	7.4 6.4	0.674 0.700	0.204 0.170	30.2 24.3
	blade	1	14.0	0.30	0.328	0.0	0.0
		2	13.0	0.16	0.304	0.0	0.0
		3	190.0 19.3	0.60 0.62	0.616 0.636	0.006 0.006	0.97 0.94
stator	base	11	32.0 34.0	11.4 8.0	1.502 1.478	0.360 0.248	24.0 16.8
		12	35.5 36.0	10.2 7.0	1.570 1/530	0.314 0.200	20.0 13.0
	blade	1	29.0 33.0 34.0	6.2 5.2 5.4	1.090 1.222 1.226	0.152 0.120 0.124	13.9 9.8 9.6
		2	30.0 25.5	5.6 6.0	1.172 1.316	0.142 0.172	12.1 13.1
		3	36.0 34.0	5.0 4.7	1.338 1.308	0.118 0.110	8.8 8.4

These measurements make it clear that the primary cause of the corrosion due damage of the blades was overheating during grinding. This is indicated primarily by the high coulombic reactivation ratios obtained for the base section of the blades (where the metal was "singed" by excessive grinding), and confirmed by microstructural examination (Figs. 5-7) as well as by the fact that the intensity of attack (the coulombic ratio) tends to fade off with increasing depth under the surface (with repetition of the EPR measurement). Grinding produced high temperatures mainly at the blade surfaces, whereas at greater depths the temperatures reached must have been lower.

The results given in Table 1 indicate that,

- generally, the "singed" areas exhibit high values of the coulombic reactivation ratios C_r/C_p, which are a warning flag attesting to the presence of an increased corrosion sensitiveness due to the precipitation at grain boundaries of chromium carbides;
- the replicated potentiodynamic measurements run at the same point of the heat-affected surface always gave lower reactivation ratio values, indicating that the "singed" surface had been removed, causing the effect of overheating to fade away as the subcutaneous layers of the steel are exposed.

Fig. 5 The outset of the corrosion crack found on the blade base.
Vilella-Bain etchant, magnif. 500.

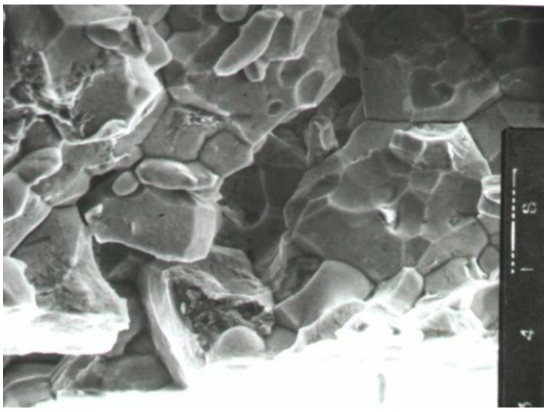

Fig. 6 Denuded surface of corrosion crack on the blade base.
In the lower part of the picture, intact blade surface.
SEM, magnif. 1000.

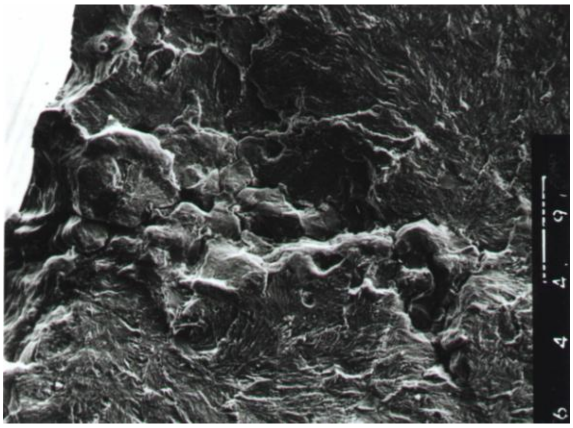

Fig. 7 Starting point of propagation of a fatigue crack on the facettes
affected by intergranular corrosion.
SEM, magnif. 1000.

Thus, coupled with metallography, the results of the electrochemical measurements indicate that:

- The cracks run a clearly intergranular course (Fig. 5), preferentially along the δ-ferrite / martensite boundaries, with occasional jerks along the martensite / martensite boundary. This has been confirmed by fractographic analysis of the denuded surfaces of the cracks (Fig. 6).
- Fatigue cracks can initiate in locations sensitized to and damaged by intergranular corrosion (Fig. 7).

In repeated potentiodynamic measurements conducted on the same surfaces after prudent polishing, this negative effect *i.e.* sensitization due to overheating, falls off gradually, due to the removal of the original (most affected) surface layer.

5 DISCUSSION

Martensitic steels such as grade 14Cr17Ni2 generally have a tendency to become sensitized to intergranular attack by a number of factors—the corrosion environment, applied mechanical loads, internal stress, etc. The same effect of the tempering temperature on the critical passivation current density J_p and the C_r / C_p ratio was observed on quenched-and-tempered martensitic 14Cr17Ni2 stainless steel (quenched at 1040°C/3min./oil and tempered for 4 h) as revealed by earlier EPR measurements conducted in 0.5M H_2SO_4 + 0.01 % KSCN at ambient temperature, at a scan rate of 15 $V{\cdot}h^{-1}$.

It is known that the temperature of 550°C is a critical temperature inducing a martensitic transformation in these materials (Fig. 8).

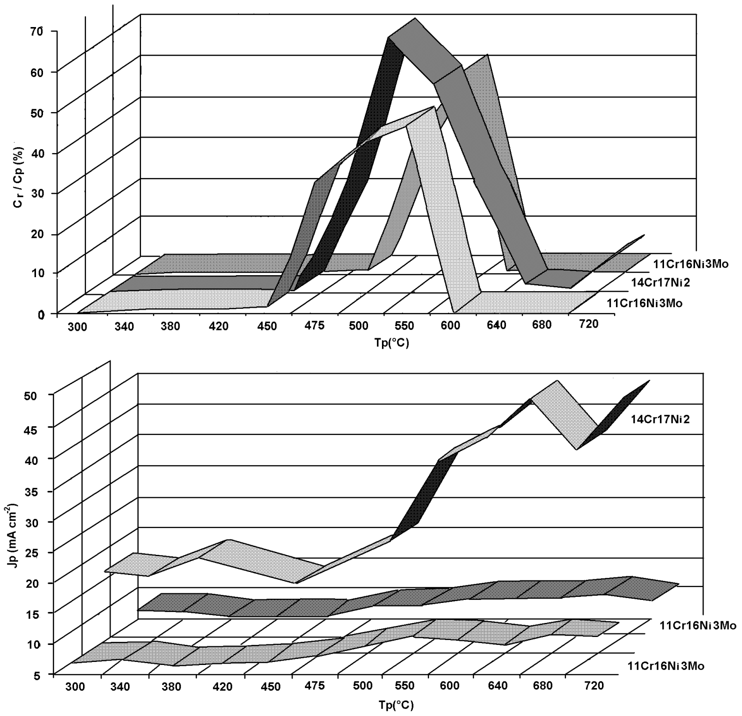

Fig. 8 Effect of tempering temperature on the coulombic ratio (top) and on the critical passivation current density (bottom) for martensitic stainless steels quenched at 1040°C. Scanned in 0.5M H_2SO_4 + 0.01% KSCN at laboratory temperature and 9 V/h scan rate.

It is typical of the classical martensitic stainless steels such as Cr17Ni2 or Cr16Ni3Mo with relatively higher carbon contents (~0.11% C) that show a critical temperingtemperature range at ca. 500°C, clearly indicated by EPR measurements of coulombic ratios (Fig. 8, top). Indeed, this quantity is indicative of grain boundary depletion of chromium, whereas the critical passivation current density measurements (Fig. 8, bottom) fail to indicate grain boundary sensitization—the only curve in the bottom figure sloping upwards reflects not sensitization but a higher degree of heterogeneity of a higher-carbon steel producing greater carbidic precipitation.

The tendency to sensitization is directly linked to the character of structure at the boundaries where ferrite δ interfaces with martensite, and the primary grain boundaries are influenced mainly by the redistribution processes involving the carbidic phase during the processing cycles which produce thermomechanical exposure. The resultant phenomena exhibit the character of temper brittleness. Susceptibility to intergranular damage is inherent in the very metallurgical design of this steel grade, and can become strengthened by certain fluctuations of the steelmaking and fabrication processes applied—also including the rather inconspicuous but despite of this, too vigorous grinding and polishing.

6 CONCLUSIONS

The study of grade 14Cr17Ni2 steel destined for compressor blades has resulted in the following conclusions:

1. The corrosion resistance of the steel is greatly reduced by thermal exposure to temperatures within the range near 500°C, causing considerable reactivation due to grain boundary sensitization to preferential attack.
2. Fatigue cracks can initiate in metal sensitized to and damaged by intergranular corrosion. They are intergranular and follow preferentially along the δ-ferrite / martensite boundaries and occasionally the martensite / martensite boundary.
3. The potential polarization reactivation method has proven to be a tool well-suited to ascertaining the optimum conditions of treatment for martensitic stainless steels and can also serve as a nondestructive measuring technique.

These results highlight the fact that in fracture mechanics studies, it occasionally pays off also to concentrate upon the corrosion effects to which the material under study is being subjected, inasmuch as both these mechanisms—fracture failure as well as corrosion attack—may act in a concerted manner and interact with each other. Thus it may be expedient to combine the interpretative capabilities of fracture mechanics and corrosion engineering.

REFERENCES

[1] International standard ISO 12732. Corrosion of metals and alloys – Electrochemical potentiokinetic reactivation measurement using the double loop method (based on Cihal's method), 2009, 15 p.

[2] V. Cihal, A. Desestret, M. Froment, G.H. Wagner, Etude de nouveaux tests potentiocinétiques de corrosion intergranulaire des aciers inoxydables, Etude CETIM—Centre de Recherches de Firminy, C.A.F.L., Rapport no. 958, December 1969

[3] V. Cihal, T. Shoji, V. Kain, Y. Watanabe, R. Stefec, EPR—A comprehensive review, Tohoku Univ., Sendai, Japan (2002) 142 p.

[4] J. Radon, Materials selection for the blades of axial turbo-exhaustors (in Czech), PhD thesis, Czech Tech. Univ., Prague, 1992, 150 p.

[5] V. Cihal, J. Hubackova, J. Kubelka, K. Mazanec, Materials Chemistry & Physics 11, 1984, p. 279

[6] V Cihal, Intergranular corrosion of steels and alloys, Elsevier-SNTL, Amsterdam-Prague 1984

Acknowledgment. This work was assisted by CR Grant agency project GACR #106/08/1789 and Ministry of Education project MSM #2579478701.