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Application of a New Device for Diagnosing the Degree of Structure Degradation of Details from Nickel-Based Superalloy

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Abstract. The possibility of applying the magnetic methods of non-destructive testing discussed with regard to details from Nickel-based superalloys. The tests based of measurement of magnetic susceptibility performed with use of the new device with improved sensitivity. The severe deformation in turbine blade after exploitation led to the appearance of a strain-induced magnetism in initially paramagnetic alloy. The strain-induced magnetism associated with formation of ferromagnetic clusters inside of the particles of intermetallic phase. The obtained magnetic effect correlated with the dynamic stress level and the number of lattice defects in various parts of the blade.

Keywords: non-destructive test, magnetic properties, deformation, Nickel-based superalloy

INTRODUCTION

Superalloy EP-800 widely used in Russian power industry [1]. Structure of this alloy consists of the nickel solid solution, 40 % of the strengthening γ'-phase (intermetallic compound Ni_3Al) and a small amount of carbides (2 %). The intermetallic compound Ni_3Al is a weak ferromagnetic with a Curie temperature $T_c = 41$ K [2]. A deviation from stoichiometry or alloying lead to increases of Curie temperature [3], but at room temperature the Ni_3Al is paramagnetic. As a result, all phases of the alloy is paramagnetic at room temperature and retain this state during its further increase. For blades of the EP-800 alloy, whose upper level is 900°C, the working temperature, as a rule, is 800°C (*standard* regime) with operation time 27000 h. In the literature, there is no information that any superalloy changed its magnetic properties after the operation on the *standard* regime during the warranty period. Magnetic methods of nondestructive testing were not in demand for Nickel-based superalloys to date.

On the other hand, some intermetallic compounds, including Ni_3Al, known to exhibit strain-induced ferromagnetism, the phenomenon in which a paramagnetic intermetallic compound becomes ferromagnetic in part upon heavy deformation [4]. Practically superparamagnetic state observed. Since it revealed no formation of any new phases, the description of the strain-induced ferromagnetism performed using the term “magnetic cluster”. Note that the above results obtained under cold deformation (for example, cold rolling). The only observation of this effect in Nickel-based superalloy ChS-70 after high-temperature deformation is [5].

The appearance of ferromagnetic ordering in the initially paramagnetic alloy results from the formation of magnetic clusters inside the cuboids of the strengthening intermetallic phase (Ni_3Al). Changing magnetic susceptibility values can be detected by an original device for the magnetic susceptibility measurements named FERROCOMPAS which was developed by the authors. The previous device IMPAS developed in this research group is currently used in industry [6–7]. New device is compact, portable, has an improved sensitivity ($\pm 1 \cdot 10^{-4}$) and working with computer for processing the measurement results. It suitable to work with low magnetic (austenitic) materials, such as stainless steel [8]. The possibility of applying this device to a three-phase material was considered in [9]. In this article, the device FERROCOMPAS applies for another class of low magnetic materials, to Nickel-based superalloys.

In this article, the degree of structure degradation of the turbine blades made from Nickel superalloy EP-800 studied by structural and magnetic methods after long-time operation with increased temperature and level of dynamic stress.

EXPERIMENT

The study of structure and magnetic properties was carried out on as-cast polycrystalline blade from the superalloy EP-800 after operation at the stationary gas turbine of an industrial type with increased temperature from 800°C in *standard* regime up to 880 °C. Operation time was 9000 h with 17 turbine starts. The purpose of the experiment was to enhance the thermal efficiency and output capability of the power generation gas turbines (GTs). The main way of increasing power is to increase the operating temperatures and rotation speed [10-11].

The chemical composition of the investigated Ni-base superalloy given in Table 1.

Magnetic tests performed at room temperature using the FERROCOMPAS device for the magnetic susceptibility measurements. Processing of results performed using calibration samples. The vibrating magnetometer Lake Shore 7407 also used for measurements of the magnetization of the samples. Measurements performed at the frequency of 82 Hz. The amplitude of vibration was 1.5 mm; the relative error of measurement was not more than 1 %.

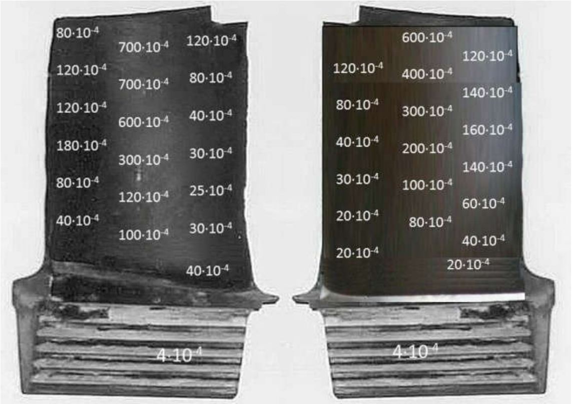
Studies of the fine structure made in the Test center of nanotechnology and advanced materials, Institute of metal physics UB RAS using a transmission electron microscope JEM-200CX, for X-rays microanalysis was used scanning electron microscope JSM 6490.

RESULTS AND DISCUSSION

After operating during 9000 h at the *experimental* regime (880°C), an increase in the magnetic susceptibility χ of blades material observed. The distribution of the magnetic susceptibility values on the surface of the turbine blade shown in Fig. 1.

The initial values of the magnetic susceptibility of superalloy EP-800 (before high-temperature deformation) was low: $\chi = 4 \cdot 10^{-4}$. The increase χ was different in different parts of the blade.

The maximum magnitudes were obtained for the convex feather part on its "back", in area where, as well known, temperature and the dynamic stress level were maximal [12]. The back of the feather is a narrow zone running along the axis of the feather on its convex side at the place of maximum curvature. Another critical place is the leading edge of the feather.



a b

Figure 1. Results of the magnetic susceptibility Measurement superimposed on the Photograph of the Blade from the both Sides: a – convex part; b – the concave part

In this case, we observe the phenomenon of deformation-induced magnetism after high-temperature deformation. This phenomenon is associated with intermetallic strengthening phase Ni_3Al, or rather with the formation of the stable defects complexes inside the γ'-phase particles. In the samples, which were cut from the convex part of the feather, the studies revealed a large number of defects in both the solid solution and in the intermetallic particles of γ'-phase (Ni_3Al) (Figure 2, a–b). The main defects were stacking fault defects within the deformed particles of intrermetallic γ'-phase (Fig. 2, c–d). The stacking faults were visible on the dark-field images. It proved that they belong to the strengthening intermetallic phase (superstructure stacking faults).

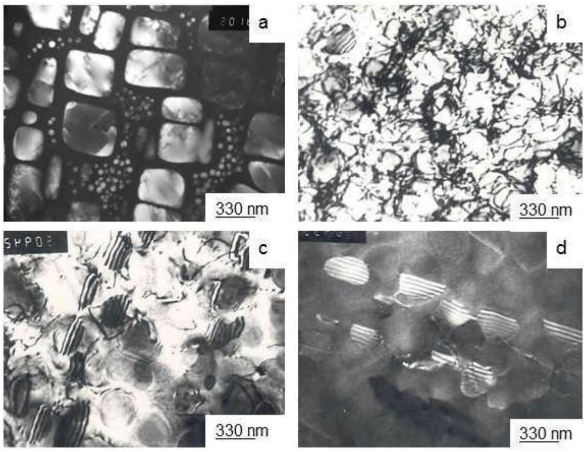


Figure 2. Structure of the different Parts of the turbine Blade after operation at 880°C for 9000 h: a – particles of intermetallic phases in the feather, dark-field image in the reflex of γ'-phase; b – high density of defects in the convex feather part on its back, the bright-field image; c, d – stacking faults inside the particles of intermetallic γ'-phase in the convex feather part on its back

In the locking turbine part, there were no defects inside γ'-particles and the only structural effect was a coagulation of intermetallic phase under heating (Fig. 3, a). The annealing of the deformed samples in a stepwise mode led to the restoration of defect-free state inside γ'-phase particles (Fig. 3, b).

Note that the electron microscopic analysis did not revealed formation of any new phase. The only phase transformation is carbide reaction when the carbide NbC replaced by carbide (Cr, Mo, W)$_{23}C_6$. These carbides are paramagnetic.

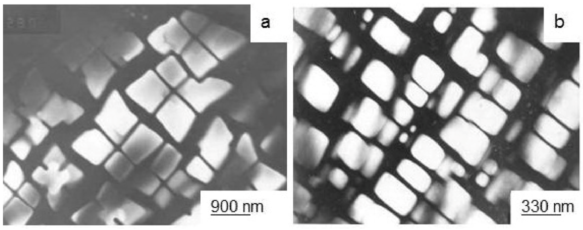


Figure 3. Intremetallic Phase, Dark-field Images in the Reflex of γ'-phase: a – locking part;
b – sample cut from the turbine blade after operation 880°C, 9900 h and reductive annealing in a stepwise mode

Thus, we see the correspondence between the number of the crystal structure defects in the various parts of the blade and the values of the magnetic susceptibility. Note the long-time operation of turbine blades from superalloys in the *standard* regime did not lead to the formation of stable defects complexes inside particles of the strengthening intermetallic phase. The defects located preferably in the solid solution. Such type of structure did not lead to the appearance of ferromagnetic properties of the paramagnetic alloy. Thus, magnetic cluster can be thought of as a complex of defects inside the intermetallic phase.

The formation of the stable defect complex inside the γ'-phase testifies of its softening. The properties of the intermetallic phase approach those of a solid solution, and the γ'-phase ceases to be strengthening component.

In this study magnetic susceptibility measurement performed directly on the blades without preliminary preparation of the surface that corresponds to the method of non-destructive magnetic testing.

The surface of blade was oxidized therefor it was necessary to trace the role of oxidation in the magnetic properties change. The high concentration of chromium (12.2 %) leads to the formation of the oxide Cr_2O_3 layer on the surface. This oxide is antiferromagnetic with low value of magnetic susceptibility. This is a protective oxide.

At first, it is necessary to establish whether there was a diffusion redistribution of alloying elements. The greatest interest to us is the area of the feather back, the place where the dynamic stress is maximal.

This zone is shown by hatching in Fig. 4 and this zone was used also for X-rays microanalysis of the alloy chemical composition. Statistical processing of data carried out according to results of measurements obtained in 12–16 points along lines marked as 1–3 at the scheme**.**

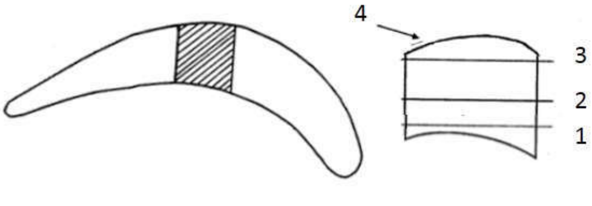


Figure 4. Scheme of X-rays microanalysis Zone on the feather cross Section

Table 1 shows the results of a study of possible redistribution of alloying elements in the alloy under the action of temperature and stress. It can conclude that the diffusion redistribution of chemical elements significantly suppressed with alloying. Our results obtained by X-rays microanalysis revealed of the redistribution of chemical elements only in two areas of the feather surface (4 at the scheme): near its back and leading edge.

Table 1. Chemical Composition of Alloy EP-800 according to the Specification and the average Composition as determined on Specimen cut from the Feather (wt. %), Carbon ≤ 0.05 wt. %

Alloying element	Cr	Mo	W	Al	Co	Nb	Ni	Fe
Specification	12.0–13.5	5.0-7.0	6.0-8.0	4.2-5.0	8.5-10.5	1.5-2.0	54.0-60.0	≤1
1	12.2	6.0	7.1	4.5	9.5	2.2	57.6	1.1
2	11.9	6.8	7.7	4.2	9.1	2.3	56.7	1.0
3	12.3	6.4	7.4	4.4	9.6	2.2	56.5	1.0

Table 2. Chemical Composition of the oxide Layer on the external Surface (4 at the Scheme)

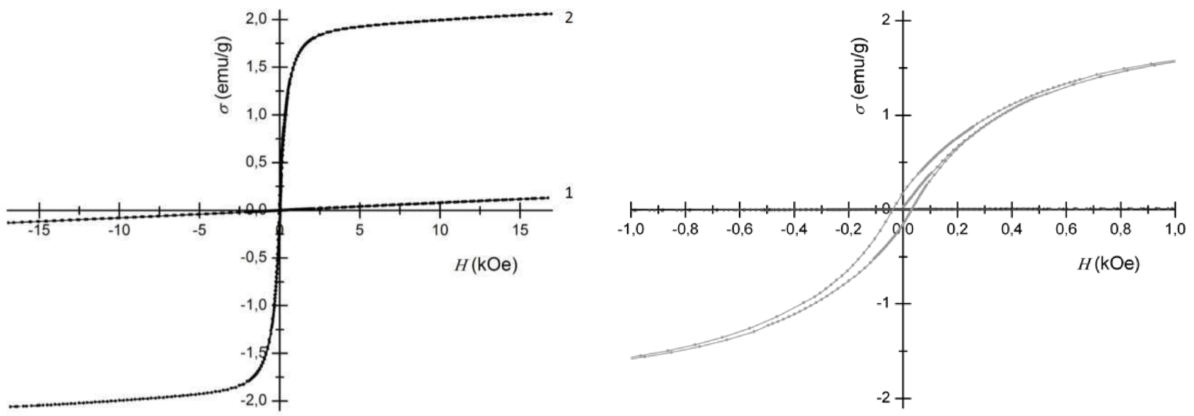
Alloying element	Cr	Mo	W	Al	Co	Nb	Ni	Fe	O	C
Oxide layer on the feather	12.6	3.7	6.5	4.1	3.3	2.2	51.1	5.6	5.2	7.9
Oxide layer on the back of the feather	4.6	3.6	1.3	7.2	3.2	2.3	43.6	6.2	13.5	16.8

It the oxide layer there was an increase in the iron concentration from 1 % up to 6 % (iron was not an alloying element and presented as an impurity) together with the decrease in the chromium content from 12.2 % down to 4.6 %. It led to the formation of ferromagnetic iron oxides. As a result, places of the blade the most prone to stress were a subject to surface corrosion. The formation of ferromagnetic iron oxide is both a contribution to the increase of the magnetic susceptibility, and the evidence of the degradation of the structure as a demonstration of diffusion under stress.

The structure degradation is real because the increases in operating time at the *experimental* regime led to the emergency destroy of the blade after 9390 h (13 months).

The independent magnetic measurements held using a vibrating magnetometer Lake Shore 7407. The measurement by vibrating magnetometer conducted by certified methods on verified device, but this method of study was destructive. The experiment carried out on the samples in the form of thin plates (0.3 mm). They were the workpiece parts of foils for the electron microscopy described above. Samples cut in such a way that they do not include oxidized surface.

Field dependence of the specific magnetization *M*(*H*) is representing in Fig. 5. The results were consistent with a previous experiment: the values of magnetic susceptibility increased in the convex feather part of the blade after operation 880 °C, 9000 h and the field dependence of magnetization represents a curve with saturation for the same sample.



a b

Figure 5. for Samples cut from different Parts of the turbine Blade after operation 880 °C, 9000 h: a – field dependence of the specific magnetization *M*(*H*): 1 – locking part; 2 – convex feather part on its back; b – the same curve *M*(*H*) on a larger scale

Hysteresis also observed, but the hysteresis loop is very narrow, suggesting the formation of super-paramagnetic state. In Fig. 5, b hysteresis loop shown for the sample cut from the blade on a different scale.

CONCLUSIONS

It is found that the initially paramagnet superalloy EP-800 acquires some ferromagnetic properties after long-time exploitation with increased operation temperature and rotation speed. The obtained magnetic tests results correlate with the dynamic stress level and the number of lattice defects in various parts of the blade.

The surface oxidation can be an additional factor of the magnetic susceptibility increase due to iron oxide formation by the diffusion under stress.

Both of these factors, and the formation of defects inside the particles of the strengthening intermetallic phase, and the formation of ferromagnetic oxides on the surface of the feather, at the same time indicate degradation of the structure.

A measurements of the magnetic susceptibility by the improved sensitivity device allow detecting the structure degradation and lead to use the magnetic nondestructive testing methods for evaluating the output capacity of turbine blades.

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