The Influence of YB Ionic Implantation on Selected Properties of TsNK-7p Alloy

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Abstract – The research concerns implantation of Yb ions into the nickel superalloy TsNK-7P. The ionic implantation has been performed using VITA installation at ions energy 30 keV. The influence of the radiation doze on depth distribution of microhardness, heat resistance and fatigue properties of the superalloy has been investigated. It has been established that the maximal effect of the implantation occurs at the radiation dose of $5 \cdot 10^{16}$ ions per square cm. This fact could be explained with the depth distribution of Yb and formation of small dispersed phases. A distinct difference between fatigue fractures of implanted and non-implanted samples has been shown. It has been established that the implantation of the superalloy at the radiation dose 5.10¹⁶ ions per square cm increases the heat resistance by 30% and fatigue properties by 11% compared to the non-implanted state. These results could be employed in the gas turbines manufacturing for increasing of their operation reliability.

1. Introduction

During recent years the heat resistance of the nickel superalloys has been increased by alloying with micro amounts of rare earth elements [1]. Introducing up to 1% of yttrium, cerium and lanthanum reduces the speed of high temperature oxidation (HTO) by 2000...2500%; this effect appears due to the increased adhesion of the oxide film to the super alloy surface [2]. Significant affinity for oxygen of the introduced elements provokes formation of the fine-grained structure of oxides, reduction in the activity of oxygen in the surface layer and increase of the diffusion rate of cations Al^{3+} and Cr^{3+} , which form stable protective layers on the surface [3].

One of the most promising methods of the surface layer microalloying is ionic implantation [4]. The creation of the effective concentration of the alloying additive is highly determined by the type of the implanted ion and by the radiation dose. The authors of Ref. [5] studied the influence of the ionic implantation of different metals on the oxidation process of titanium. They established that the highest HTO resistance is reached after implantation of the ions of the rare-earth elements that possess the higher values of formation heat for their stable oxides. The results of the studies in Ref. [6] which concern the influence of the ytterbium ions implantation regimes on pure nickel HTO show that with the radiation dose $(D) \le 6 \cdot 10^{17}$ ions per square cm the heat resistance of the nickel samples rises and with $D > 10^{18}$ ions per square cm – decreases. The authors associate data obtained with the concentration of ytterbium in the surface layer and the surface irregularities, which appear as a result of ion-beam dispersion.

The purpose of this publication is (i) study of the influence of the ionic implantation of ytterbium on the physical and chemical state of the surface layer and on the heat resistance of the TsNK-7P superalloy, (ii) selection of the effective radiation dose, which increases in the operating characteristics of the superalloy.

2. Experimental

The implantation of samples was performed on the installation VITA at ions energy 30 keV, ion current density 20-40 µA per square cm and the radiation doses of 10^{16} ... $2 \cdot 10^{17}$ ions per square cm. For study the heat resistance, the samples in the initial and irradiated states tested at 850 °C for 300 hrs in accordance with ALL-UNION STATE STANDARD 6130-71. For determination of the modified layer depth, microhardness has been measured on slanting sections (load 20 g, instrument PMT-3). The depth distribution of the implanted element in has been investigated using secondary ion mass spectroscopy on the installation POLUS-4. Change of the surface chemical activity has been evaluated by measuring the electrode potential of surface (AgCl electrode, 5% NaCl solution). The limit of fatigue strength was determined in accordance with ALL-UNION STATE STANDARD 25.502-81 on base of $N = 2 \cdot 10^7$ cycles at 600 °C on the vibrostand VEDS-400A. The comparative analysis of fractures was conducted on scanning electron microscope JXA-6400 (JEOL).

3. Results and Discussion

For the purpose of preliminary select of effective radiation dose, the theoretical concentration profile of the ytterbium, implanted with the energy 30 keV into the pure nickel has been calculated. The calculation has been performed in accordance with the theory of Linkhard, Scarf and Shiott, taking into account the possible dispersion the implanted surface [5]. It was established that with $D = 10^{16}$ ions per square cm the concentration depth distribution of the Yb ions could be expressed by a Gaussian curve with the position of maximum at the depth of 4 ... 5 nm. At $D = 5 \cdot 10^{16}$ ions per square cm, the maximal concentration of ytterbium is observed on the surface, remaining constant up to depth of $3.5 \dots 4$ nm. Further, it monotonically goes to the zero value at depth of $17\dots 18$ nm. The subsequent increase in the dose of implantation does not change the profile of ytterbium concentration. This limits the possible range of radiation dose with the current energy of the implanted ions.

The theoretical assumptions have been validated experimentally on the samples made from TsNK-7P superalloy. The results are presented in Figs. 1–3. The research shows that the mass-spectra contain two peaks that prove the presence of ytterbium and oxides Yb_xO_y in the surface layer. The analysis of change in the relative intensity of the peaks in the dependence on the radiation dose (Fig. 1) shows that the maximum intensity of the peaks occurs at $D = 5 \cdot 10^{16}$ ions per square cm. The less radiation doses do not provide the maximal saturation of the surface layer by ytterbium ions, whereas with the radiation doses of more than $D = 5 \cdot 10^{16}$ ions per square cm, in addition to the ionic saturation, the dispersion of the surface takes place.

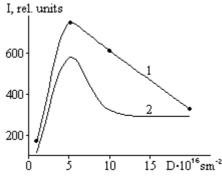


Fig. 1. Change in the relative intensity of the peaks of ytterbium (1) and oxide Yb_xO_y (2) in the dependence on the radiation dose

The presence of the ytterbium oxides in the implanted layer has also been confirmed by the measurements of the electrode potentials of the alloy surface. The oxides of ytterbium, which possess covalent bond, provide the surface passivation and shift the electrode potential into the less negative region compared to the initial state (Fig. 2, curve 1). The strongest passivation has been observed with samples at $D = 5 \cdot 10^{16}$ ions per square cm. With the increase in the radiation dose to $D = 2 \cdot 10^{17}$ ions per square cm, where the effect of dispersion occurs, the growth of the roughness leads to the surface activation. Therefore, the absolute value of the electrode potential reduces.

The change of ytterbium concentration in the alloy depending on the radiation dose also affects the depth of the modified layer. The results of measuring the microhardness on the slanting sections with the load of 20 g (Fig. 3, curve 1) and the average microhardness with the use of a direct method with the load 50 g (Fig. 3, curve 2) shows that the maximum strain hardening of the superalloy surface is also observed with $D = 5 \cdot 10^{16}$ ions per square cm. The depth of the modified layer that have increased microhardness considerably exceeds the depth of projective ion path [7].

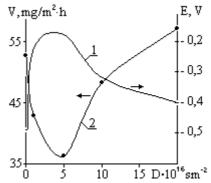


Fig. 2. Change in the value of the electrode potential of surface (1) and rate of oxidation of TsNK-7P superalloy at 850 °C (2) in the dependence on the radiation dose

This phenomenon is connected with formation and evolution of radiation defects. The flow of interstitial atoms into the depth of the sample leads to the creation of complex dislocatory structure. The distortions of the crystal lattice strengthens the superalloy [8].

The interaction of the ion beam with the surface of the superalloy also changes the heat resistance. The ions of ytterbium implanted into the alloy play the role of additional crystallization centers. As a consequence, the rate of the protective oxide film formation increases.

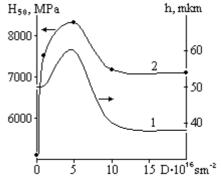


Fig. 3. TsNK-7P superalloy microhardness (1) and depth of modified layer (2) change in the dependence of the radiation dose

The data represented in Fig. 2 (curve 2) show that the influence of radiation dose on the oxidation resistance appear to be ambiguous. The heat resistance rises at $D \le 10^{17}$ ions per square cm and reaches the maximal value at $D = 5 \cdot 10^{16}$ ions per square cm. With the increase in the radiation dose more than $D > 10^{17}$ ions per square cm, the positive effect of the implantation is reduced. At these conditions the heat resistance of the implanted and non-implanted samples is almost identical, since the dispersion of surface increases the roughness of the surface of the irradiated models and the area of the oxidized surface goes up.

Finally, $D = 5 \cdot 10^{16}$ ions per square cm appear to be the most effective radiation dose for modifying the surface of the TsNK-7P nickel superalloy.

The change in composition and microstructure of the surface and near-surface layers of the superalloy by ionic implantation also increases the fatigue strength of the superalloy [5]. The results of tests for cyclic fatigue strength at the temperature of 600 °C are presented in Fig. 4. The tests show the positive influence of the Yb ionic implantation of surface. It has been established that the limit of the fatigue strength of the superalloy that is irradiated by the dose of $5 \cdot 10^{16}$ ions per square cm rises by 11% compared to the non-irradiated state.

Fractographic analysis shows distinct features in the appearance of samples fractures in the initial and implanted states after the fatigue temperature tests. In both cases the destruction has multi-focus, sub-surface character. In the samples with the initial surface state the morphology of fracture has spalling on the crystal faces. In the implanted samples the destruction begins on the crystal planes within one-two grains. Then the cracks extend throughout the grain bodyies by the mechanism of viscous diffusive destruction with the formation of the grooves of fatigue [9].

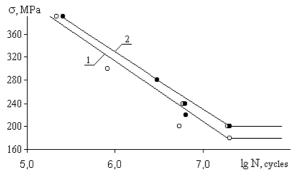


Fig. 4. Curves of fatigue strength at the test temperature 600 °C: 1 – TsNK-7P superalloy, 2 – TsNK-7P superalloy implanted by the dose of $D = 5 \cdot 10^{16}$ ions per square cm

This difference is connected with the fact that the fine dispersed phases which contain ytterbium serve as an effective barrier in the path of motion of dislocations. They prevent dislocations confluence and development of microscopic cracks [10]. This mechanism provides the large cyclic longevity of the implanted samples.

4. Conclusions

The implantation of the TsNK-7P superalloy with ytterbium ions considerably changes the properties of the surface layer. The maximal depth of the ionic-modified layer and the highest heat resistance could be reached at the radiation dose of $5 \cdot 10^{16}$ ions per square cm. Application of this dose of implantation increases the fatigue strength of superalloy by 11% and, as a result, the operational reliability of details.

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