

Anisotropy of Mechanical Properties of Single Crystal in Fourth Generation Ni-Based Superalloy

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Abstract

Single crystals of Ni-based superalloys have significant anisotropy of mechanical properties. Data about anisotropy of Young modulus, ultimate tensile strength, yield strength, elasticity, and rupture strength of Russian single crystal Ni-based superalloys of fourth generation with $\langle 001 \rangle$, $\langle 011 \rangle$, $\langle 111 \rangle$ crystallographic orientation are presented in article.

1. Introduction

In contrast to polycrystalline isotropic materials mechanical properties of single crystals of Ni-based superalloys with face-centered cubic structure are significantly anisotropic and depend on crystallographic directions, along which these properties are defined [1]. For structural strength analysis of single crystal blades and life-time projecting of GTE it's necessary to have data about temperature and orientation dependence of physical-mechanical properties in superalloys.

Vital information, mentioned in this article, shows fundamental mechanisms of temperature and orientation dependence of Young modulus and characteristics of short-time strength (ultimate tensile strength and yield strength, elongation and reduction area), strength rupture of Russian single crystal Ni-based superalloy N4 of fourth generation with $\langle 001 \rangle$, $\langle 011 \rangle$, $\langle 111 \rangle$ crystallographic orientations [2, 3].

2. Materials and data processing technique

The chemical composition of the investigated single crystal superalloy № 4 is given in Table 1 [2].

Table 1: Chemical composition single crystal superalloy N4, wt.%

| Al | Cr | Mo | W | Ta | Co | Re | Ru | Ni |
|-----|-----|-----|-----|-----|-----|-----|-----|---------|
| 6.0 | 3.3 | 4.0 | 4.0 | 5.0 | 7.0 | 6.5 | 4.0 | Balance |

Specimens for mechanical tests (gage length – 25 mm, diameter – 5 mm) were manufactured from round single crystal bars, which axes coincide within 10 degrees of the crystallographic directions $\langle 001 \rangle$, $\langle 011 \rangle$ or $\langle 111 \rangle$. Single crystal bars (\varnothing 16 mm and length 185 mm) were produced by the LMC method (Liquid Metal Cooling). The $\langle 001 \rangle$, $\langle 011 \rangle$, $\langle 111 \rangle$ growth was initiated by a seed of a high temperature Ni-W alloy placed in a special cavity of the ceramic mould. The single-crystals grown under given solidification conditions have a typical dendritic-cellular macrostructure with the primary dendrite arm spacing of about 250 μm . Thermal treatment, including step-by-step homogenization in 1285–1320 $^{\circ}\text{C}$ temperature range for 26 hours and two-step aging at 1130 and 870 $^{\circ}\text{C}$ was performed in order to form optimal microstructure (Fig. 1).

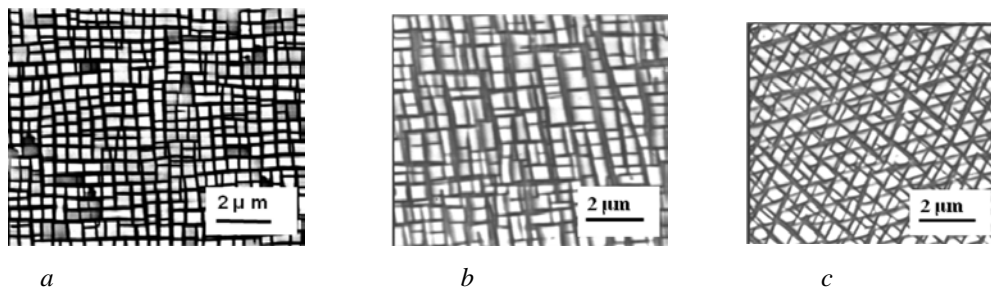


Figure 1: The γ/γ' -microstructure in the single crystals superalloy N4 after full heat treatment (cross-section):
a – $\langle 001 \rangle$ orientation; *b* – $\langle 011 \rangle$ orientation; *c* – $\langle 111 \rangle$ orientation

During homogenization first the nonequilibrium eutectic $\gamma+\gamma'$ dissolves and then the chemical composition within a dendritic cell homogenizes. Solution of the $\gamma+\gamma'$ eutectic results in the formation of micropores in the interdendritic regions. These homogenization pores have typical size of about 10 μm . After full heat treatment the differences in γ' -shape and size in the dendrite axes and interdendritic regions become smaller. Over the whole dendritic cell the γ' -precipitates have cuboidal morphology and size of 0,4-0,5 μm .

Characteristics of short-time strength, elasticity and Young modulus of $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ single-crystals of the superalloy N4 were measured using MTS-810 machine at a strain rate of 1 mm/min at temperature range of 20–1150 $^{\circ}\text{C}$.

$\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ single-crystals of the superalloy N4 were tested under creep conditions at temperatures 750, 850, 950 and 1150 $^{\circ}\text{C}$ (within the range of tolerance $\pm 10^{\circ}$) until rupture. The results of performed tests were fitted by equation [4]:

$$\tau = \xi T^m \sigma^{-n} \exp\left(\frac{U_0 - \eta \sigma}{RT}\right) \quad (1)$$

where τ is creep lifetime in hour; σ stress in MPa; T temperature in K; ξ , m , n , U_0 (activation energy in $\text{J}\cdot\text{mol}^{-1}$), η (activation volume in $\text{J}\cdot(\text{mol}\cdot\text{MPa})^{-1}$) adjustable parameters; R – universal gas constant in $\text{J}\cdot(\text{K}\cdot\text{mol})^{-1}$. Using the obtained values of adjustable parameters the levels of rupture stress (σ_r) were calculated for lifetimes $\tau = 100$ and 1000 hours at 750, 850, 950 and 1150 $^{\circ}\text{C}$.

3. Experimental results and discussion

Figure 2 shows diagrams of Young modulus vs. temperature in single crystals with $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ crystallographic orientations. While increasing test temperature from 20 up to 1150 $^{\circ}\text{C}$, Young modulus monotonically decreases. The single crystals with $\langle 111 \rangle$ crystallographic orientation have maximum value of Young modulus and $\langle 001 \rangle$ the single crystals – the minimum value. Intermediate values of Young modulus are typical for $\langle 011 \rangle$ single crystals. It should be mentioned, that the significant anisotropy of Young modulus remains in all investigated temperature interval.

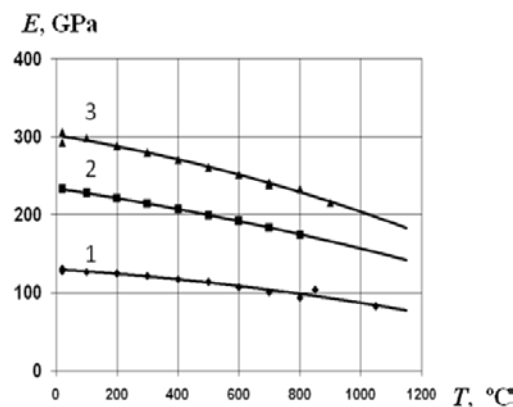


Figure 2: Temperature dependence of Young modulus in single crystals superalloy № 4 of $\langle 001 \rangle$ (1), $\langle 011 \rangle$ (2), and $\langle 111 \rangle$ (3) orientations

The anisotropy of short-time strength properties in single crystals is defined by the specific slip systems in the existing experimental conditions. Since plastic deformation behavior is defined as interaction of slip systems, tensile test diagrams will be different for different orientation of the single crystals [5-7].

The temperature dependences of ultimate and yield strengths of single crystals with the three tested crystallographic orientations are shown on Fig. 3. In 20–700 $^{\circ}\text{C}$ temperature range, the single crystals with $\langle 001 \rangle$ and $\langle 011 \rangle$ orientations, have a small temperature dependence of ultimate tensile strength and yield tensile strengths. On further increasing of temperature one can see increasing of these parameters up to maximum at peak temperature of 800 $^{\circ}\text{C}$, then alloy softening is proceeded at more elevated temperatures. Such changing of ultimate strength and yield strengths in two-phase single crystal Ni-based superalloys at elevated temperatures can be explained using the abnormal temperature dependence of yield stress in particles of intermetallic γ' -phase [1]. With respect to single crystals with $\langle 111 \rangle$ crystallographic orientation, monotonic decrease of the ultimate and yield strengths in all analyzed temperature range is typical. It should be mentioned, that yield strength of the Re-Ru containing single crystals superalloys fourth generation has lower values, than the Re-containing single crystals superalloys 3rd

generation. Similar ratio of ultimate strength and yield strength is observed for single crystals of Re-containing superalloy PWA 1484 and Ru-containing superalloy EPM-102 [8].

The reduced yield strength is typical characteristic of single crystals in Re-Ru-containing superalloys. It can be associated with the influence of Ru on the dislocation mechanism of plastic deformation.

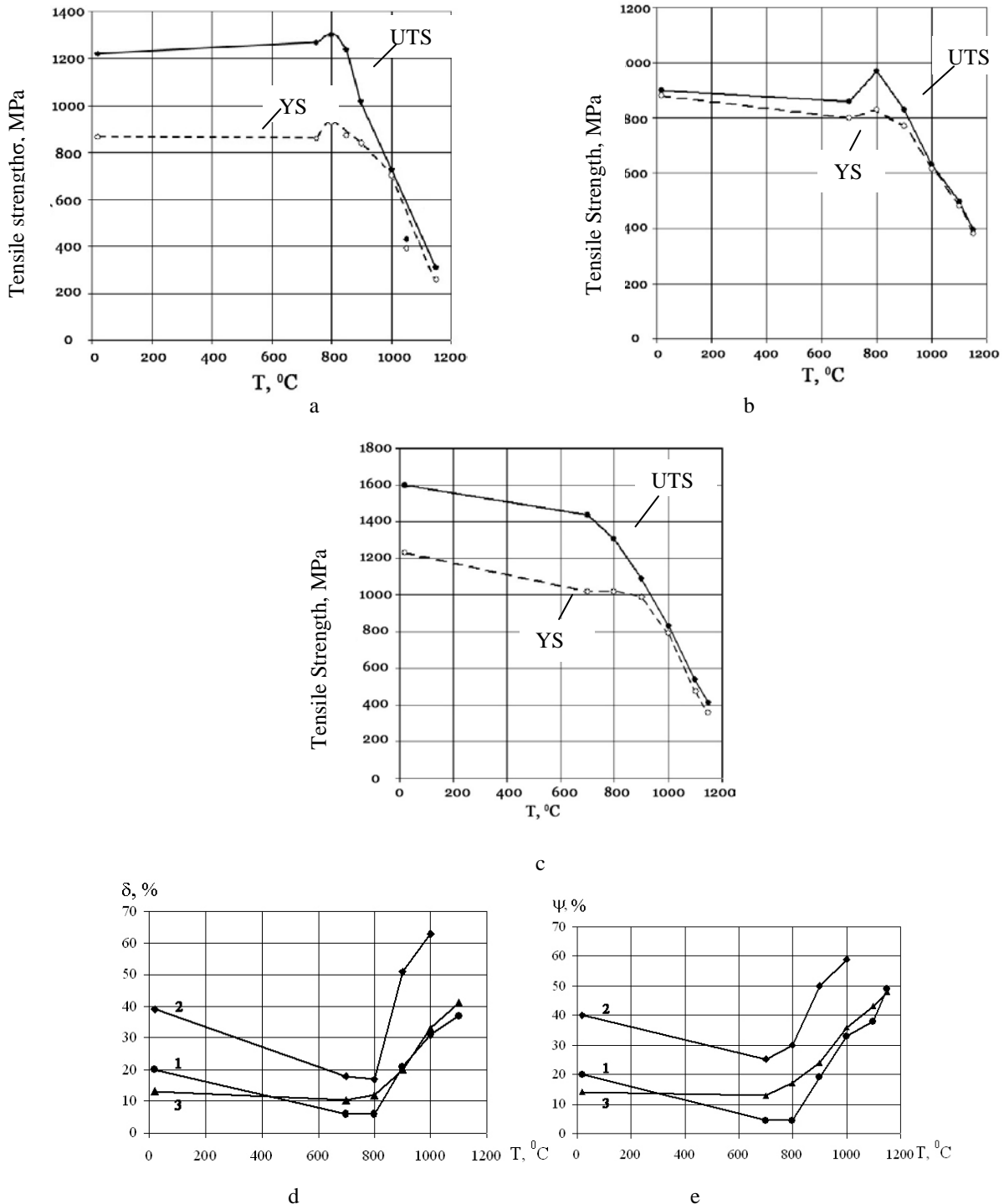


Figure 3: Temperature and orientation dependence of ultimate tensile strength, 0.2% yield tensile strengths (a – $\langle 001 \rangle$ orientation; b – $\langle 011 \rangle$ orientation; c – $\langle 111 \rangle$ orientation), elongation δ (d) and reduction area ψ (e) of $\langle 001 \rangle$ (1), $\langle 011 \rangle$ (2) and $\langle 111 \rangle$ (3) in the single crystals superalloy № 4.

The temperature dependence of the elongation and reduction area in the single crystals with the three crystallographic orientations are shown on Fig. 3de. On increasing the temperature, both of these properties decrease

to a minimum at peak temperature. At temperatures, higher than the peak, significant increasing of elongation and reduction of area was observed.

Table 2 shows rupture strength for 100 and 1000 hours, calculated using experimental data, for three crystallographic orientations of single crystals superalloy № 4 in 750–1150 °C temperature range. For estimation of anisotropy of stress rupture it's suitable to use coefficient [9]:

$$K_{\tau}^{\text{hkl}} = \frac{\sigma_{\tau}^{\text{hkl}}}{\sigma_{\tau}^{\text{001}}} \quad (2)$$

where $\sigma_{\tau}^{\text{hkl}}$ – stress rupture of single crystal with $\langle \text{hkl} \rangle$ crystallographic orientations for τ hours, $\sigma_{\tau}^{\text{001}}$ – stress rupture of single crystal with $\langle 001 \rangle$ crystallographic orientations for τ hours.

Table 2. Stress rupture for 100 and 1000 hours of $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$ single crystals superalloy № 4 and coefficients of anisotropy

| Temperature, °C | Rupture strength for 100 hours, σ_{100} , MPa | | | K_{100}^{011} | K_{100}^{111} | Rupture strength for 1000 hours, σ_{1000} , MPa | | | K_{1000}^{011} | K_{1000}^{111} |
|-----------------|--|-----------------------|-----------------------|-----------------|-----------------|--|-----------------------|-----------------------|------------------|------------------|
| | $\langle 001 \rangle$ | $\langle 011 \rangle$ | $\langle 111 \rangle$ | | | $\langle 001 \rangle$ | $\langle 011 \rangle$ | $\langle 111 \rangle$ | | |
| 750 | 1015 | 690 | - | 0.67 | - | 835 | 616 | - | 0.73 | - |
| 850 | 725 | 485 | 800 | 0.67 | 1.10 | 565 | 395 | 640 | 0.78 | 1.13 |
| 950 | 405 | 340 | 540 | 0.83 | 1.33 | 265 | 240 | 360 | 0.90 | 1.35 |
| 1150 | 125 | 102 | 115 | 0.82 | 0.92 | 74 | 77 | 65 | 1.04 | 0.87 |

As it can be seen from the table, coefficient K_{100}^{011} is less than 1 in 750–1150 °C temperature rang, i. e. stress rupture for 100 hours for $\langle 011 \rangle$ single crystals is less, than for $\langle 001 \rangle$ single crystals. Stress rupture of $\langle 111 \rangle$ in 850–950 °C temperature range is more, than for $\langle 001 \rangle$ single crystals, as K_{100}^{111} is more than 1. But at temperature of 1150 °C coefficient of anisotropy K_{100}^{111} is less than 1, i. e. $\langle 111 \rangle$ single crystals are softening more intensive and their heat resistance is less, than for $\langle 001 \rangle$ single crystals. Similar dependence is observed while long-run tests for 1000 hours.

Irreversible changes of alloy phase structure and morphology of strengthening γ' -phase particles is preceded while high-temperature creep in the structure of single crystals. Such changes are visually illustrated by the evolution of voluntary unit microstructure, enclosed with (001) planes, in single crystal specimen $\langle 001 \rangle$ of initial state and after high-temperature creep at temperatures of 1000 and 1100 °C (Fig. 4).

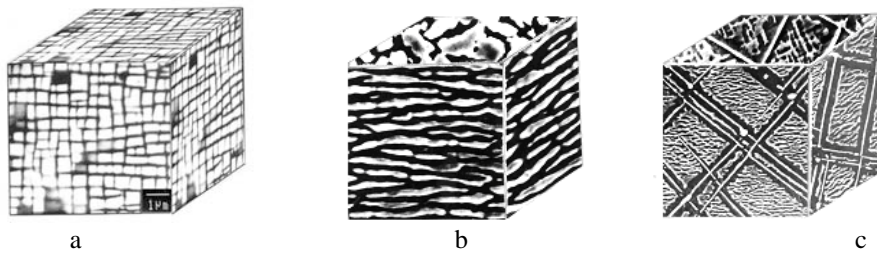


Figure 4: Voluntary unit microstructure of $\langle 001 \rangle$ single crystals superalloy № 4 after heat treatment (a), creep tests at 1000 °C, $\sigma=200$ MPa test period – 1274 h (b), and 1100 °C, $\sigma=120$ MPa test period – 1303 h (c)

As it can be seen from Fig. 4a, alloy voluntary unit microstructure in initial conditions consists of γ -solid solution matrix, where cuboidal particles of γ' -phase have tendency to ordered arrangement, forming quasiperiodic three-dimensional macro lattice. Such microstructure is morphologically unstable at high temperature creep tests. In transition phase of high temperature creep initial cuboidal particles of γ' -phase are specifically coagulated at the expense of plate splicing. Broad facets of plates are perpendicular to load axis (raft-structure) (Fig. 4b).

Regular raft-structure of single crystals make active reaction on to plastic creep deformation at high temperatures, blocking γ' -phase plates intersection. Dislocations migrate in interlayers of γ -solid solution. In this connection any disturbance of raft-structure promotes softening and decreasing of stress rupture.

Phase instability in single crystals of analyzed alloy is discovered in creep process in 1000–1100 °C temperature range. Phase instability is visualized in initiation of lamellate particles, Re inreached (TCP phases). TCP phases are isolated in first order dendrite axes in $\langle 111 \rangle$ plates. These plates transverse with voluntary unit cube face in $\langle 110 \rangle$ crystallographic direction (Fig. 4c). Volume concentration and geometry (thickness and length) of TCP phase plates increase while increasing test period at 1100 °C. Lamellate isolations in single crystal structure are not visualized after creep tests at 1150 °C during 3000 hours test period.

One of mechanisms of Ni-based superalloys stationary creep at elevated temperatures and low tension provides for nonconservative motion of dislocations along interface of γ'/γ plates in raft -structure. Supernumerary vacancies, condensed in pores of deformation origin, were generated [10]. These pores were visualized in single crystals at 1000, 1100 and 1150 °C and low tension, i.e. at 1000 hours test period. Pores are located in interaxial areas at lineages of blocks (Fig. 5a). Unique feature of such pores is contained in cuboidal morphology and enclosing $\langle 110 \rangle$ plates. Such enclosure is stipulated because of significant anisotropy of surface tension at 1000–1100 °C test temperatures.

Pores in the single crystals are stress raisers, and micro cracks are initiated while tertiary creep. Micro crack initiation originates either on coarse homogenized spherical form pores, or fine deformation pores, generated while high temperature creep in wide test period. It depends on temperature and test period.

At temperatures, more than 1000 °C and low stress i.e. long rupture time micro crack initiation take place by deformation pores (Fig. 5b). Micro cracks initiate in interdendritic areas. Microcrack coalescence in most damaged area reduces to a main crack propagation and final specimen destruction.

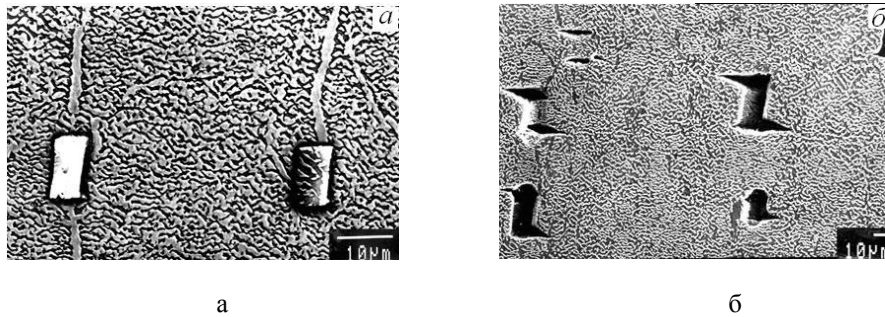


Figure 5: Morphology of deformation pores at lineages of blocks in specimen volume (a), microcrack nucleation on deformation pores (b)

4. Conclusions

1. The Youngs modulus anisotropy remains in all temperature range 20–1150 °C, $\langle 111 \rangle$ single crystals superalloy № 4 have the maximum value of Young modulus, $\langle 001 \rangle$ single crystals – minimum.
2. Significant anisotropy of ultimate strength and yield strength is observed only down to 800 °C. The anisotropy almost degenerates up to this temperature; i. e. crystals begin to soften with the same speed. At 800 °C strength properties of $\langle 001 \rangle$ and $\langle 011 \rangle$ single crystals are at peak values, plasticity properties – minimum. Monotonic decreasing of strength is typical for $\langle 111 \rangle$ single crystals when increasing test temperature in 20–1150 °C range.
3. Significant stress rupture anisotropy for single crystal of three orientations is observed, $\langle 111 \rangle$ single crystals have maximum value of heat resistance, $\langle 011 \rangle$ single crystals – minimum. Stress rupture anisotropy is almost degenerated at 1150 °C.

5 References

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