Original paper

Surface modification of TiNi-based alloys by high-current low-energy electron beams

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Abstract. Titanium nickelide alloys *TiNi* exhibit a high sensitivity of their fine crystalline structure to external energy inputs, which governs their functional properties (the shape-memory effect and superelasticity). Exposure to high-current low-energy electron beams produces metastable nanocrystalline states in the near-surface layers; however, the quantitative analytical relationship between the integral irradiation energy and the evolution of structural parameters remains insufficiently developed. Based on X-ray diffraction data and computer simulation of heat transfer, a physical and mathematical model has been developed that describes the kinetics of changes in the unit-cell parameter a, second-kind microstrains $\varepsilon = \langle \Delta d/d \rangle$, and coherent scattering region sizes D_{CSR} as functions of the integral energy $W = N \cdot E_S$. Exponential saturation of the decrease in the lattice parameter $a(W)$, with a limiting contraction $\Delta a/a_0 \approx 0{,}69\%$ caused by selective titanium evaporation, has been established. The dependences $\varepsilon(W)$ and $D_{CSR}(W)$ were obtained and approximated by threshold functions reflecting the competition between the defect generation and annealing over different activation-energy spectra. The dislocation density $\rho = (1{,}5\text{-}16{,}7)\cdot 10^{14}$ m^{-2} and elastic energy of the defect subsystem $U_{disl} \approx 4{,}0 \cdot 10^7$ J/m^3 were calculated. The proposed model predicts the structural state of the modified layer with an accuracy of $R^2 > 0{,}96$ and elucidates the physical mechanisms governing threshold changes in the fine crystalline structure, thereby providing a basis for targeted control of the functional responses of shape-memory alloys.

Keywords: titanium nickelide, high-current electron beams, X-ray diffraction analysis, lattice parameter, thermodynamic modeling.

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Модификация поверхности сплавов на основе TiNi высокоточным низкоэнергетическим электронным пучком

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Аннотация: Сплавы на основе никелида титана *TiNi* характеризуются высокой чувствительностью тонкой кристаллической структуры к внешним энергетическим воздействиям, что определяет их функциональные свойства (эффект памяти формы,

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сверхэластичность). Воздействие низкоэнергетических сильноточных электронных пучков формирует в приповерхностных слоях метастабильные нанокристаллические состояния, однако количественная аналитическая связь между интегральной энергией облучения и эволюцией структурных параметров остаётся недостаточно разработанной. На основе данных рентгеноструктурного анализа и компьютерного моделирования теплопереноса разработана физико-математическая модель, описывающая кинетику изменения параметра элементарной ячейки a, микроискажений второго рода $\varepsilon = <\Delta d/d>$ и размеров областей когерентного рассеяния D_{CSR} в зависимости от интегральной энергии $W = N \cdot E_S$. Установлено экспоненциальное насыщение снижения параметра решётки $a(W)$ с предельным сжатием $\Delta a/a_0 \approx 0{,}69\%$, обусловленное селективным испарением титана. Получены зависимости $\varepsilon(W)$ и $D_{CSR}(W)$, которые аппроксимированы пороговыми функциями, отражающими конкуренцию генерации и отжига дефектов с различным спектром энергий активации. Рассчитаны плотность дислокаций $\rho = (1{,}5\text{-}16{,}7)\cdot 10^{14}$ м$^{-2}$ и упругая энергия дефектной подсистемы $U_{disl} \approx 4{,}0 \cdot 10^7$ Дж/м^3. Предложенная модель обеспечивает прогноз структурного состояния модифицированного слоя с точностью $R^2 > 0{,}96$ и раскрывает физические механизмы порогового изменения тонкой кристаллической структуры, что создаёт основу для адресного управления функциональными откликами сплавов с памятью формы.

Ключевые слова: никелид титана, сильноточные электронные пучки, рентгеноструктурный анализ, параметр решётки, термодинамическое моделирование.

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1. Introduction

Pulsed concentrated energy fluxes (laser, ion, and electron beams) provide ultrafast heating-cooling cycles (10^6-10^9 K/s), leading to the formation of metastable nanocrystalline and amorphous phases with modified physico-mechanical characteristics [1-3]. Currently, surface modification of metals and alloys is quite widely used upon irradiation with high-energy electron beams [4, 5]. Thus, in [6], data are presented on the evolution of the structure and properties of the surface layer of an industrial steel subjected to irradiation with a high-intensity pulsed electron beam of submillisecond duration in the melting mode and subsequent high-speed crystallization of the modified layer. Research is being actively conducted on the effect of irradiation with an intense pulsed electron beam on the transformation of the structure and phase composition of the surface layer of hypereutectic and hypoeutectic silumins [7, 8].

The $NiTi$ intermetallic compound is a benchmark material exhibiting the shape-memory effect and superelasticity; its macroscopic properties are governed by the martensitic transformations $B2 \rightleftharpoons B19'$, which are critically sensitive to the stoichiometry, unit-cell parameter, and crystal-lattice defect density [9]. Despite the accumulated experimental evidence [10-13], a quantitative description of the evolution of the structural parameters of the $NiTi$ near-surface layer under exposure to high-current low-energy electron beams remains fragmentary. No unified analytical model is available that relates the integral irradiation energy W to the lattice parameter a, microstrains ε, and coherent scattering region size D_{CSR}, while accounting for the threshold nature of the dislocation-substructure transformation and selective evaporation of the alloy constituents.

The aim of this study is to develop a physical and mathematical model by means of computer simulation, introducing analytical dependences and thermodynamic estimates verified by numerical examples based on X-ray diffraction data on the effect of high-current low-energy electron beams on changes in the near-surface layers of a titanium-nickelide-based alloy.

2. The object of the research

The object of investigation was a titanium-nickelide-based alloy of the $Ni_{49,7}Ti_{50}Cu_xMo_{0,3}$ composition. The alloys were melted at the Research Institute of Medical Materials and Shape-Memory Implants. The melting quality was controlled by weighing the charge and the ingots. Melting was performed in an inert-gas atmosphere (argon). The resulting ingots were rolled into strips of 1,1 mm thick. Rolling was carried out at temperatures of 800-900°C. The alloyed titanium-nickelide strips were then descaled by etching in a solution of nitric and hydrofluoric acids. Specimens with dimensions of 1×1×45 mm were cut from the prepared strips.

Experiments on irradiating the alloy surface with a low-energy, intense pulsed electron beam were conducted under the following parameters: the electron beam energy density per pulse E_S varied from 2 J/cm^2 to 7 J/cm^2, and the pulse duration was constant τ = 1,5 μs. The number of pulses varied from N = 1 to N = 50 at a repetition rate of f = 10 s^{-1}.

X-ray diffraction studies were performed using an XRD-6000 Shimadzu diffractometer with $Cu_{K\alpha}$ radiation and a Ni filter. Extrapolation plots $a = f(\cos^2\theta)$ were used to determine the unit-cell parameter of the B2 phase, which reduced the influence of systematic errors in the precision determination of the lattice parameter. Phase identification was performed from the positions and intensities of the Bragg reflections. The appearance of additional weak lines was regarded as evidence of secondary-phase precipitation or the formation of a

martensitic constituent.

3. Results and discussion

X-ray diffraction analysis revealed that the primary phase of the alloy in its initial state is the austenite phase with a B2 structure. The secondary phase, present in small quantities, is the Ti_2Ni phase. Their intensity is low and therefore corresponds to a small amount of secondary phases. After irradiation of the alloy surface with the low-energy intense pulsed beam, no enhancement of the structural-reflection intensities of the secondary phases was detected in the diffraction patterns.

The X-ray diffraction data are presented in Fig. 1-3. Analysis of these data revealed a systematic decrease in the unit-cell parameter of the austenitic B2 phase with increasing integral irradiation energy, which correlates with the evaporation of titanium atoms from the surface (see Fig. 1). The observed experimental decrease in the lattice parameter of the $TiNi$ matrix phase with the B2 structure during pulsed electron-beam irradiation of the sample surface is attributed to the following processes. At the initial moment of interaction, the electron beams from the first pulses lead to heating of the sample surface to temperatures on the order of 2100 K and melting of the surface layer up to ~3 μm thick. Thus, the temperature created on the sample surface exceeds the melting temperature of the $TiNi$ matrix phase ($T_m(TiNi) \approx 1580$ K). The fine-dispersed Ti_2Ni particles that were present in the $TiNi$ matrix phase dissolve in the melt, leading to local titanium enrichment. This is followed by ultrafast crystallization at a cooling rate of $\sim 5\times10^8$ K/s, which results in the 'freezing-in' of a nonequilibrium state in which Ti atoms do not have time to diffuse back into the particles. During subsequent pulses or during cooling, precipitation of ultrafine-dispersed Ti_2Ni particles occurs, leading to depletion of the $TiNi$ matrix phase in titanium.

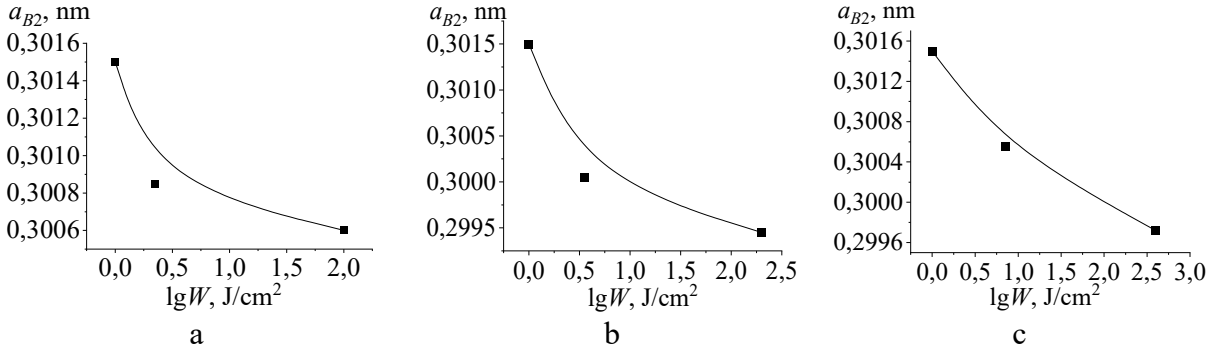

Fig. 1. Dependences of the lattice parameters on the integral beam energy, a – pulse energy E_S = 2 J/cm^2; b – 4 J/cm^2; c – e = 7 J/cm^2.

The dependences of the second-kind microstrains $\langle \Delta d/d \rangle$ and coherent

scattering region sizes D_{CSR} are nonmonotonic, indicating a threshold character of the structural evolution (see Fig. 2, 3). Above the threshold energy, the beam modifies the spectrum of existing defects: dislocation configurations with a low activation energy of annealing annihilate, whereas defects with a high activation energy accumulate. Electron-microscopy data confirm the transformation of the dislocation substructure from a banded disoriented morphology to a network morphology as the integral energy input increases.

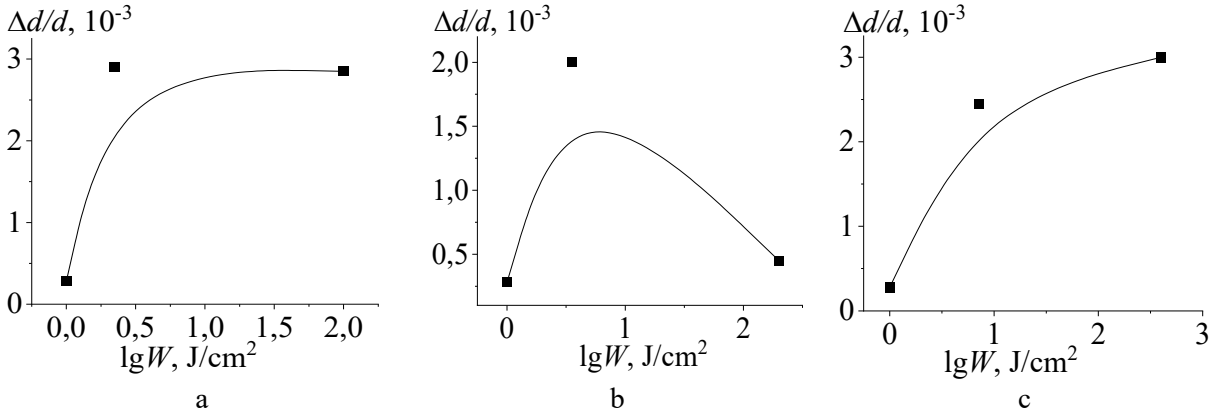

Fig. 2. Dependences of the second-order microstresses on the integral beam energy, a – pulse energy E_S = 2 J/cm^2; b – 4 J/cm^2; c – e = 7 J/cm^2.

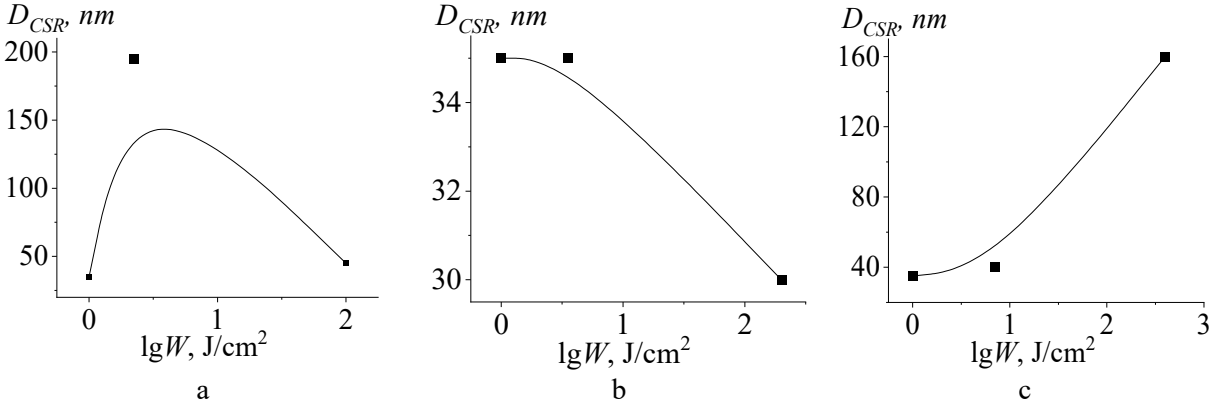

Fig. 3. Dependences of the coherent scattering region on the integral beam energy (a – pulse energy E_S = 2 J/cm^2; b – 4 J/cm^2; c – e = 7 J/cm^2.

4. Computer Simulation of Heat Transfer

Thermophysical processes were modeled by solving a non-stationary heat conduction equation, taking into account phase transitions and surface losses

$$\rho c_p \frac{\partial T}{\partial t} = \nabla \cdot (k \nabla T) + Q(z,t) - \sigma\varepsilon\left(T^4 - T_0^4\right) - h_c\left(T - T_0\right) \quad (1)$$

where $Q(z,t) = I_0(1-R)\alpha e^{\alpha z} f(t)$ is the volumetric source of absorbed power, R is the reflection coefficient, α is the absorption coefficient, σ is the Stefan-Boltzmann constant, ε_S is the surface emissivity, and h_c is the convective heat-transfer coefficient. The simulation results are as follows. The molten-layer

thickness is $h_{melt} = 3{,}0 \pm 0{,}2$ μm. The maximum surface temperature is $T_{\max} \approx 2100$ K (i.e. higher than $T_m^{NiTi} \approx 1580$ K). The calculation showed that the characteristic cooling rate is ~$5 \cdot 10^8$ K/s.

This rate exceeds the critical value for suppressing diffusive redistribution of atoms (~10^5 K/s), thereby ensuring the «freezing-in» of the high-temperature defect structure. The fine crystalline structure was investigated by X-ray diffractometry ($Cu_{K\alpha}$ radiation, $\lambda = 0{,}15418$ nm). The grain-size and microstrain contributions were separated using the modified Williamson-Hall method [14].

Analysis of the data shown in Fig. 1 revealed a monotonic decrease in the lattice parameter a of the $B2$ phase with increasing integral electron-beam energy W. The calculation shows that the maximum relative decrease in the lattice parameter of the $B2$ phase in the near-surface layer, i.e., the contraction, reaches values on the order of $\Delta a / a_0 \approx$ -$0{,}7 \cdot 10^{-3}$ (0,7%).

Analysis of the microstrain dependence $\varepsilon(W)$ reveals a pronounced maximum at intermediate energies ($\lg W \approx 0{,}35$-$0{,}82$), after which ε decreases (see Fig. 2).

Fig. 3 reveals a complex dependence $D_{CSR}(W)$: an initial increase to 200 nm, subsequent fragmentation to 40 nm, and coalescence to 175 nm at the maximum values of W. These trends indicate a change in the dominant mechanisms upon reaching threshold values of the integral irradiation energy. The decrease in the unit-cell parameter a is interpreted within a model of selective titanium evaporation from the melt surface. For a binary alloy, the parameter change is described by the generalized Vegard law [9, 15]:

$$\alpha\left(x_{Ti}\right) = a_0 + \beta\left(x_{Ti} - 0{,}5\right) + \gamma\left(x_{Ti} - 0{,}5\right)^2 \qquad (2)$$

where x_{Ti} is the mole fraction of titanium, $a_0 = 0{,}3015$ nm is the lattice parameter of the initial alloy before irradiation, $\beta \approx 0{,}018$ nm/at. fr., and $\gamma \approx$ -0,012 nm/at. fr^2 [9]. The evaporation-rate component is proportional to the saturated vapor pressure

$$P_{sat}\left(T\right) \propto \exp\left(-L_{vap} / k_B T\right). \qquad (3)$$

Under pulsed heating, the integral mass loss follows a power law

$$\Delta m \propto W^{v}\left(v \approx 0{,}8 - 1{,}0\right). \qquad (4)$$

Approximation of the experimental data demonstrates exponential saturation:

$$a\left(W\right) = a_\infty + \left(a_0 - a_\infty\right)\exp\left(-\frac{W}{W_a}\right) \qquad (5)$$

where W_a is the characteristic lattice-relaxation energy. Based on the calculation in the linear approximation of the Vegard dependence of the lattice parameter on concentration, it was established that:

$$\Delta x_{Ti} \approx \frac{-0,00697 \cdot 0,5}{0,018 / 0,3015} \approx -0,058 . \quad (6)$$

Thus, the titanium content in the near-surface layer decreases by ~5,8 at.%, which agrees with thermodynamic estimates of evaporation at $T \approx 2000$ K [16].

Second-kind microstrains ε are related to the dislocation density ρ by [17]:

$$\varepsilon = \frac{b}{2\pi}\sqrt{\rho \cdot C_M} \quad (7)$$

where $b = a\sqrt{2} \approx 0,213$ nm is the Burgers vector for dislocations with the $-\langle 111 \rangle$ direction in the $B2$ lattice, and $C_M = 1,5 \div 2,0$ is the dislocation contrast factor accounting for their type and orientation relative to the scattering vector. Hence, the expression for calculating the dislocation density is

$$\rho(W) = \left(\frac{2\pi\varepsilon(W)}{b \cdot C_M} \right)^2 . \quad (8)$$

Calculations based on Eq. (8) show that the dislocation density in the near-surface layer may reach values on the order of $1,67 \times 10^{-15}$ m^{-2}.

The elastic energy of the dislocation subsystem per unit volume is estimated as [18]:

$$U_{disl} = \frac{Gb^2\rho}{4\pi(1-\nu)} \ln\left(\frac{R}{r_0}\right). \quad (9)$$

Calculations using Eq. (9) and the parameters from [1] showed that $U_{disl} \sim 3,95 \cdot 10^7$ J/m^3.

To approximate the nonmonotonic behavior of $\varepsilon(W)$, we use a kinetic model describing the competition between defect generation and annealing:

$$\varepsilon(W) = \varepsilon_{\max} \left(\frac{W}{W_\varepsilon} \right)^n \exp\left[n\left(1 - \frac{W}{W_\varepsilon} \right) \right] \quad (10)$$

where n is the curve-steepness parameter and E_ε is the position of the microstrain maximum. For the $e = 7$ J/cm^2 series, nonlinear regression gives $e_{\max} = 2,8 \cdot 10^{-3}$, $E_\varepsilon = 8,2$ J/cm^2, and $n = 1,4$ ($R^2 = 0,983$). The observed evolution of $D_{CSR}(W)$ and the change in substructure types (banded → network → cellular) are described by the distribution of defects over annealing activation energies $g(E_a)$. The concentration of defects surviving after N pulses is calculated as

$$C(W) = \int_{E_{\min}}^{E_{\max}} q(E_a) \exp\left[-N\nu_0 t_{eff} \exp\left(-\frac{E_a}{k_B T_{peak}} \right) \right] dE_a \quad (11)$$

where $\nu_0 \approx 10^{13}$ s^{-1} is the attempt frequency for overcoming the barrier, τ_{eff} is the effective annealing time during the pulse, and T_{peak} is the peak temperature. The

approximate analytical solution leads to a threshold dependence of the coherent scattering region size:

$$D_{CSR}(W) = D_0 + \Delta D \cdot \left[1 - \exp\left(-\frac{W}{W_{th}}\right)\right] \cdot \exp\left(-\frac{W}{W_{sat}}\right) \quad (12)$$

where W_{th} is the threshold for the onset of subgrain coalescence, and W_{sat} is the saturation energy of the fragmentation process. The calculated size of the regions D_{CSR} in the irradiated specimens is approximately 174 nm. The discrepancy from the experimental value (40 nm) is caused by a change in the fragmentation mechanism at $W > W_{fr} \approx 80$ J/cm^2, which is accounted for by adding the contribution $-\Delta D_2\left[1 - \exp\left(-W - W_{fr}\right)/W_{fr2}\right]$.

A thermodynamic analysis is performed on the basis of the change in the Gibbs free energy of the modified layer relative to the initial state, represented as the sum of the contributions

$$\Delta G(W) = \Delta G_{chem} + \Delta G_{el} + \Delta G_{gb} + \Delta G_{surf}\,. \quad (13)$$

The contributions were calculated, and their numerical values are presented in Table 1.

Table 1. Numerical values of contributions to the Gibbs free energy.

N	Contribution	Calculation formula and numerical value of the contribution
1	Chemical	$\Delta G_{chem} = \int_{x_0}^{x(W)} \mu_{Ti}(x)\,dx \approx 1,2 \cdot 10^6$ J/m^3
2	Elastic	$\Delta G_{el} \approx U_{disl} + \frac{1}{2} B\varepsilon^2 \approx 4,1 \cdot 10^7$ J/m^3
3	Grain-boundary	$\Delta G_{gb} = \frac{3\gamma_{gb}}{D_{CSR}} \approx 3,38 \cdot 10^7$ J/m^3
4	Surface	$\Delta G_{surf} = -\sigma_{rel} W$, $\sigma_{rel} \approx 0,8$ J/(m^3 · J/cm^3)

Analysis of the total balance of contributions to the Gibbs free energy shows that at electron-beam energies $W < 50$ J/cm^2, ΔG_{gb} dominates and stabilizes the nanocrystalline state. At $W > 100$ J/cm^2, the negative contribution ΔG_{surf} compensates for the increase in ΔG_{el}, providing the thermodynamic possibility of coalescence and a decrease in ε.

5. Conclusion

Analysis of the results made it possible to distinguish three stages in the evolution of the structural state in the near-surface region of the titanium-nickelide-based alloy under exposure to high-current low-energy electron beams.

The first stage corresponds to the low-energy regime ($W < 10$ J/cm^2), in

which surface heating occurs without through-thickness melting. Active generation of point defects and dislocations is observed at this stage. The microstrain ε increases while $D_{CSR} \approx D_0$ is retained. The kinetics of defect accumulation dominates ($d\rho / dW > 0$).

At the second stage, a threshold melting-crystallization regime is observed ($10 < W < 80$ J/cm^2). A molten layer ($h \approx 3$ μm) forms and subsequently undergoes ultrafast crystallization. A submicrocrystalline structure develops with a maximum of ε and a minimum of D_{CSR} because dislocation motion is blocked by the boundaries of newly formed grains.

The third stage is a high-energy annealing regime ($W > 80$ J/cm^2). Repeated thermal exposure causes annealing of defects with activation energies $E_a < 1{,}2$ eV. Subgrain coalescence occurs (D_{CSR}), and ε decreases, while the altered lattice parameter a is retained because of irreversible selective titanium evaporation. The dislocation substructure evolves into a regular network configuration with minimization of the elastic energy. The integral-energy parameter W serves as a universal coordinate in the space of structural states.

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