

Microstructure and Mechanical Properties of Ni₃Al-Based Single Crystal Alloy IC21^{*}

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Abstract

A low-density, low-cost, high-strength Ni₃Al-based single crystal alloy IC21 has been developed to address the service performance requirements of high-pressure turbine guide vanes. The Re content of this alloy is not more than 1.5%, and its density is less than 8.0 g/cm³. The incipient melting temperature of the alloy measured by metallographic method is approximately 1345 °C. After standard heat treatment, the γ' phase in the IC21 alloy is uniformly distributed with a volume fraction of approximately 80% and a size of about 420 nm, exhibiting a high degree of γ' phase cuboidization and ordering. The IC21 single crystal alloy exhibits a tensile strength of 490 MPa and a yield strength of 470 MPa at 1100 °C, with a stress rupture life reaching 170.5 h under conditions of 1100 °C and 140 MPa, and 110.0 h under conditions of 1150 °C and 100 MPa. The IC21 single crystal alloy possesses good high-temperature microstructural stability and excellent high-temperature oxidation resistance; after long-term thermal exposure at 1080 °C, no topologically close-packed (TCP) phases precipitated, and the oxidation kinetics curves in air at 1100 and 1150 °C for 100 h follow a parabolic law, with oxidation weight gain rates of 0.015 and 0.045 mg/(cm² · h), respectively. Microstructural analysis indicates that the high-temperature strength of this single crystal alloy primarily originates from the high γ' phase content, high lattice misfit, and dense interfacial dislocation network structure.

Full Text

Microstructure and Mechanical Properties of Ni₃Al-Based Single Crystal Alloy IC21*

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Abstract In view of the service-performance requirements for high-pressure turbine guide-vane blades, a Ni₃Al-based single crystal alloy IC21 with low density, low cost, and high strength was developed. The Re content of this alloy is not greater than 1.5%, and its density is less than 8.0 g/cm³. The incipient melting temperature of the alloy measured by metallography is about 1345 °C. After standard heat treatment, the γ' phase in the IC21 alloy is uniformly distributed, with a volume fraction of about 80% and a size of about 420 nm, and has a relatively high degree of cuboidal morphology and ordering. At 1100 °C, the tensile strength and yield strength of the IC21 single crystal alloy are 490 MPa and 470 MPa, respectively. Under conditions of 1100 °C and 140 MPa, its stress-rupture life reaches 170.5 h; under conditions of 1150 °C and 100 MPa, its stress-rupture life reaches 110.0 h. The IC21 single crystal alloy has good high-temperature microstructural stability and good high-temperature oxidation resistance. After long-term thermal exposure at 1080 °C, no topologically close-packed phase pre-

precipitates. In air at 1100 and 1150 °C for 100 h, the oxidation kinetic curves follow a parabolic law, with oxidation weight-gain rates of 0.015 and 0.045 mg/(cm² · h), respectively. Microstructural analysis indicates that the high-temperature strength of this single crystal alloy mainly originates from the high γ' -phase content, the large alloy misfit, and the dense interfacial dislocation network.

Key words Ni₃Al, IC21 single crystal alloy, low cost, low density, high-temperature mechanical properties, oxidation resistance

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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Ni₃Al-BASED SINGLE CRYSTAL ALLOY IC21

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ABSTRACT According to the requirement of high-pressure turbine guide vane during service, the aim of this work is to design a single crystal Ni₃Al-based alloy named IC21 with low density, low cost, and high strength which can be used as high-pressure turbine guide vane material. The mass fraction of the Re has been limited less than 1.5% on purpose. The single crystal bars of IC21 were prepared by high rate solidification method. The density of IC21 is 8.0 g/cm³ and the incipient melting temperature was identified by metallography. After standard heat treatment, the distribution of the γ' precipitates is uniform with the average size of about 420 nm, and volume fraction of 80%. The tensile and yield strengths at 1100 °C are 490 and 470 MPa, respectively. Moreover, IC21 shows superior creep properties, the stress-rupture life at 1100 °C, 140 MPa is 170.5 h and at 1150 °C, 100 MPa still remains 110.0 h. The microstructure stability of IC21 alloy at 1080 °C for as long as 1000 h were evaluated. The results show that no precipitated phase exists during thermal exposure at 1080 °C, which exhibits good stability. The oxidation kinetic curves of IC21 alloy follows a parabolic rate law in different oxidation stage during cycle oxidation for 100 h in air. IC21 alloy has a good high temperature oxidation resistance, the strengthening mechanism are attributed to high volume fraction of γ' phase, large negative misfit and well-established interface networks.

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KEY WORDS Ni₃Al, IC21 single crystal alloy, low cost, low density, high temperature strength, high temperature oxidation resistance

Single-crystal high-temperature alloy turbine blades have been one of the major technological advances in aero-engines since the 1980s, making important contributions to increasing the thrust-to-weight ratio of aero-engines. At present, almost all advanced high-performance aero-engines use single-crystal alloy turbine blades[¹⁻³]. For high-pressure turbine blades, the temperature-bearing capability and cost of single-crystal superalloys are of crucial importance for developing aero-engines with high thrust-to-weight ratios. To improve the temperature-bearing capability of single-crystal superalloys, large amounts of refractory metal elements that provide solid-solution strengthening and precipitation strengthening are usually added, such as Re, W, Mo, Ta, and Ru[⁴⁻⁶]. The addition of these alloying elements markedly improves the temperature-bearing capability of single-crystal superalloys, but also increases alloy density and cost. For example, the third-generation single-crystal superalloy CMSX-10 has a temperature-bearing capability exceeding 1100 °C, but contains about 6% Re; the total amount of refractory metal elements approaches 20%, the density reaches 9.05 g/cm³, and the cost is about 50% higher than that of second-generation single-crystal superalloys. In addition, as the thrust-to-weight ratio of aero-engines continues to increase, thin-walled hollowing has also become one of the structural design features of high-pressure turbine blades[⁷⁻⁹]. The structural characteristics of variable cross-sections and thin-walled curved surfaces in complex single-crystal alloy hollow blades impose increasingly stringent requirements on alloy castability and single-crystal integrity.

In view of the service-performance requirements of high-pressure turbine guide vanes, and with the goal of developing a single-crystal superalloy with low cost, low density, high temperature-bearing capability, and good castability, this study developed a single-crystal superalloy material for high-pressure turbine guide vanes with a temperature-bearing capability of 1100 °C, density not greater than 8.0 g/cm³, and a cost about one half that of second-generation single-crystal superalloys.

1 Experimental Methods

The nominal composition of the master alloy used in the experiment (mass fraction, %) was: Al 7.6–8.3, Cr 1.5–2.5, Mo 9–13, Re 0.5–1.5, Ta 2.4–4.0, Y 0.01, balance Ni. The single-crystal alloy was prepared by the spiral grain-selection method. The orientation of the single-crystal specimens was determined by Laue back-reflection, and specimens deviating by less than 12° from the $\langle 001 \rangle$ direction were selected for mechanical-property tests. Phase analysis was performed using a D/max-2000PC X-ray diffractometer (XRD). The heat-treatment schedule of the alloy was determined by orthogonal experiments as: 1315°C , 4 h + 1320°C , 6 h + 1325°C , 4 h, air cooling (A.C.) + 1120°C , 2 h, A.C. + 870°C , 32 h, A.C. The microstructures of the alloy in the as-cast state, after heat treatment, after long-term aging, and after stress rupture were observed using an APOLLO-300 field-emission scanning electron microscope (SEM). The etchant used was $\text{CuSO}_4 : \text{HCl} : \text{H}_2\text{O} = 1 : 5 : 5$ (volume ratio), and the attached backscattered electron (BSE) imaging system and energy-dispersive spectroscopy (EDS) were used for composition analysis. At the same location in regions far from the fracture-necking zone, transverse (perpendicular to the stress-axis direction) and longitudinal (parallel to the stress-axis direction) samples were taken. After double-jet electropolishing and thinning (the electrolyte was 10% perchloric acid + 90% ethanol solution, volume fraction; the solution temperature was about -20°C), microstructural observation and selected-area electron diffraction (SAED) analysis were carried out using a JEM-2100F transmission electron microscope (TEM). The isothermal oxidation experiment was conducted in accordance with HB5258-2000.

2 Experimental Results

2.1 Microstructure and Microstructural Stability

Figures 1a and b are respectively the BSE images of the transverse and longitudinal sections of the as-cast IC21 single-crystal alloy specimen; Figures 1c and d are enlarged images of one dendrite arm and interdendritic region in Figure 1a, respectively. Combined with EDS analysis, it can be seen that the as-cast microstructure consists of a gray-white network-like γ phase, a black γ' phase, and white precipitated phases. The large black blocks in Figure 1c are Al-rich large γ' phases formed at the end of solidification, all located between dendrites. The white precipitates that separate out along the black large γ' phase between dendrites are Mo- and Re-rich phases. The atomic ratio of Mo to Ni is close to 1 : 1, and the composition is close to that of the Ni-Mo phase reported in the literature[10–12]. This is because precipitation of the Al-rich large γ' phase between dendrites increases the relative contents of Mo and Re in the interdendritic region, thereby forming the white Ni-Mo precipitated phase. The sizes of γ' phases in the dendrite cores are relatively uniform, whereas the interdendritic γ' phases are respectively large black γ' phases and ultrafine γ' phases much

Fig. 1

Figure 1: Fig. 1

Fig. 2

Figure 2: Fig. 2

smaller than the normal γ' phases in dendrite cores. The ultrafine γ' phases nucleate in the Al-depleted regions formed around the interdendritic large γ' phases; because the driving force for growth is smaller than that of the γ' phases in dendrite cores, they do not readily grow.

Four sets of solution heat-treatment parameters were designed to determine the solution-treatment schedule of the alloy: S1: 1315 °C, 16 h; S2: 1320 °C, 16 h; S3: 1315 °C, 4 h + 1320 °C, 4 h + 1325 °C, 6 h; S4: 1315 °C, 4 h + 1320 °C, 4 h + 1330 °C, 6 h. S1 and S2 did not achieve complete solutioning, whereas S3 and S4 both achieved complete solutioning. Statistical analysis showed that the average sizes of the γ' phases in S3 and S4 were about 400 and 460 nm, respectively. Considering the final size of the γ' phase after solution treatment comprehensively, S3 was selected as the solution-treatment schedule for the IC21 alloy. On the basis of complete solution treatment, the effect of different primary aging schedules on the microstructure of IC21 was investigated. Figure 2^[13] shows the microstructure of the IC21 single-crystal alloy after solution treatment at 1315 °C for 4 h + 1320 °C for 6 h + 1325 °C for 4 h, A.C., followed by primary aging at different temperatures for 2 h, A.C., and finally secondary aging at 870 °C for 32 h, A.C. The aging-treatment temperature affects the size, morphology, and number of γ' phases. Aging treatment of single-crystal superalloys is usually carried out in two stages. Generally, primary aging is aging at a relatively high temperature, with the purpose of adjusting the size of the γ' phase, and it has a relatively large effect on the size, number, and morphology of the γ' phase in the alloy; secondary aging is aging at a relatively low temperature, with the purpose of adjusting the cuboidal degree of the γ' phase^[14-19], and on...

Fig. 1 BSE images of the transverse and longitudinal sections of the as-cast microstructure of IC21 single-crystal alloy

Fig. 1 Cross- (a) and longitudinal- (b) sectional BSE images of as-cast IC21 single crystal alloy and partially enlarged views of Fig. 1a (c) and Fig. 1b (d)

Fig. 2 SEM images of IC21 single-crystal alloy after different aging treatments^[13]

Fig. 2 SEM images of IC21 single crystal alloy after different aging treatment^[13]

(a) 1060 °C, 2 h + 870 °C, 32 h (b) 1080 °C, 2 h + 870 °C, 32 h

Fig. 3 SEM images of IC21 alloy after thermal exposure at 1080 °C for different times: 50 h (a), 100 h (b), 300 h (c), 500 h (d), 800 h (e), and 1000 h (f).

Figure 3: Fig. 3 SEM images of IC21 alloy after thermal exposure at 1080 °C for different times: 50 h (a), 100 h (b), 300 h (c), 500 h (d), 800 h (e), and 1000 h (f).

(b) 1100 °C, 2 h + 870 °C, 32 h (d) 1120 °C, 2 h + 870 °C, 32 h

The influence of the size and number of the γ' phase is relatively small. It can be seen from Fig. 2 that, with an increase in the primary aging temperature, after secondary aging treatment the cuboidal degree of the γ' phase in the alloy increases slightly. Taking into comprehensive consideration the cuboidal degree and size of the γ' phase in the alloy after heat treatment, as well as the width of the γ channels, the final aging schedule of the alloy was determined to be 1120 °C, 2 h + 870 °C, 32 h. After standard heat treatment, the volume fraction of the γ' phase (the black phase in Fig. 2) in the IC21 single-crystal alloy was about 80%, and the size of the γ' phase was 420 nm.

The relationship between the microstructure and properties of the alloy is very close. Long-term high-temperature service environments can lead to degradation of the alloy microstructure, thereby affecting the properties of the alloy. Therefore, studying the microstructural stability of the material at high temperature is of great significance. Fig. 3^[13] shows SEM images of the IC21 single-crystal alloy after long-term thermal exposure at 1080 °C for different times. It can be seen that, after thermal exposure at 1080 °C for 300 h, the alloy can still basically maintain the microstructure in the heat-treated state. After thermal exposure for 500 h, the γ' phase grows by connecting along mutually perpendicular directions, and several neighboring γ' phases begin to coalesce. With increasing aging time, the microstructure begins to form irregularly shaped γ' phases, such as long strips and L-shaped morphologies; at the same time, the γ channels also coarsen to a certain extent with increasing time. No precipitation was observed during the long-term thermal-exposure experiment at 1080 °C.

Fig. 3 SEM images of IC21 alloy after thermal exposure at 1080 °C for 50 h (a), 100 h (b), 300 h (c), 500 h (d), 800 h (e) and 1000 h (f)^[13]

phase analysis, it can be seen that the alloy has good microstructural stability at 1080 °C^[20].

2.2 Mechanical properties

The tensile properties of the IC21 single-crystal alloy at different temperatures are shown in Table 1. The results show that the IC21 single-crystal alloy not only has very high yield strength and tensile strength, but also exhibits very

good ductility. It can be seen from the table that the yield strength and tensile strength of the IC21 single-crystal alloy increase gradually with increasing temperature, reaching maxima near 850 °C; after reaching the peak values, as the temperature continues to rise, the yield strength and tensile strength of the IC21 single-crystal alloy gradually decrease. With increasing temperature, the elongation of the alloy reaches its minimum at 760 °C. Figure 4^[21] shows the yield strength and elongation of CMSX-4 and IC21 single-crystal alloys at different temperatures. It can be seen that the overall ductility of the alloy is good; the IC21 single-crystal alloy can still maintain relatively high strength at 1100 °C, with a yield strength of 470 MPa, and the yield strength at 1150 °C is 330 MPa. Comparison with the tensile-property data for the second-generation Ni-based single-crystal superalloy CMSX-4^[21] shows that the tensile strength of the IC21 single-crystal alloy is comparable to that of the CMSX-4 superalloy. When the temperature exceeds 900 °C, the alloy's advantage in high-temperature strength begins to appear, and the variation of elongation with temperature is basically the same.

Table 2 shows the stress-rupture property data of the IC21 single-crystal alloy under medium- and high-temperature conditions. From the Larson-Miller curve of the IC21 single-crystal alloy, regression analysis gives the following equation for the stress-rupture strength parameter:

$$\sigma = 7986.55762 - 502.47935P + 8.03714P^2 \quad (1)$$

$$R = 0.992 \quad (2)$$

where σ is stress; $P = T(20 + \lg t)/10^3$, t is the stress-rupture life of the alloy, T is temperature, and R is the correlation coefficient. The correlation coefficient is as high as 0.992, indicating that the equation has very good correlation.

Figures 5a and b are TEM images of the IC21 single-crystal alloy after thermal exposure at 1100 °C for 1 h, taken at different magnifications^[13,27]. From Fig. 5a, it can be seen that after 1 h of thermal exposure of the IC21 single-crystal alloy, complete interfacial dislocation networks with square-lattice structures have formed in most regions of the alloy, indicating that the IC21 alloy has a relatively high γ/γ' interfacial misfit^[13,22]. Diffraction analysis shows that the interfacial dislocation network is mainly composed of dislocation lines with Burgers vectors $b = [101]$ and $b = [01\bar{1}]$, and that the directions of the dislocation lines in the network are along the $\langle 001 \rangle$ directions. After thermal exposure for 10 h, the interfacial dislocation network throughout the observed region has been fully established, and the structure of the interfacial dislocation network does not change significantly. After thermal exposure for 100 h, the interfacial dislocation network structure remains the same as that after thermal exposure for 1 and 10 h. The results of Zhang et al.^[23,24] and Yang et al.^[25] on the relationship between misfit and creep properties show that, as the absolute

value of the misfit increases, the spacing of the interfacial dislocation network in the alloy decreases; that is, the spacing of the γ/γ' interfacial dislocation network is inversely proportional to the γ/γ' interfacial misfit of the alloy^[26]:

$$d = b/|\delta_m| \quad (3)$$

where d is the spacing of the interfacial dislocation network, b is the magnitude of the Burgers vector, and δ_m is the alloy misfit. The average spacing of the interfacial dislocation network of the IC21 single-crystal alloy after thermal exposure at 1100 °C for 100 h is 30–32 nm; therefore, the misfit of the alloy at 1100 °C can be estimated to be approximately -0.8% . Meanwhile, using X-ray diffraction combined with fitting by the Origin peak-separation software, the misfit of the heat-treated IC21 single-crystal alloy at room temperature was measured to be about -0.38% . The above results show that, whether at room temperature or at 1100 °C, the IC21 single-crystal alloy has relatively high

Table 1 Tensile properties of IC21 single-crystal alloy at various temperatures^[13]

Temperature / °C	σ_b / MPa	$\sigma_{p0.2}$ / MPa	δ / %	Φ / %
RT	812	784	25.2	22.2
	886	834	15.2	16.9
760	1050	980	12.0	37.3
	995	911	12.4	38.7
850	1017	954	51.2	40.2
	937	889	73.2	46.5
980	690	670	41.5	39.0
	685	650	55.0	49.5
1100	490	470	33.0	64.0
	505	498	35.0	71.5
1150	350	330	47.0	78.0
	350	330	44.0	74.5

Note: RT—room temperature, σ_b —tensile strength, $\sigma_{p0.2}$ —yield strength, δ —elongation, Φ —area reduction.

mismatch.

Summarizing the above test results, the low-Re single-crystal alloy IC21 possesses a high γ' -phase volume fraction (about 80%) and a high γ/γ' interfacial misfit (up to -0.8% at 1100 °C), which are important reasons for its excellent high-temperature stress-rupture properties^[13,27]. At the same time, after stress-free thermal exposure at 1100 °C for 1 h, a dense γ/γ' interfacial dislocation network is established in the alloy; moreover, the dislocation lines in the

Fig. 4 Influence of temperature on yield strength and elongation of CMSX-4 and IC21 single crystal alloys

Figure 4: Fig. 4 Influence of temperature on yield strength and elongation of CMSX-4 and IC21 single crystal alloys

network are oriented along the $\langle 001 \rangle$ direction, and thus can significantly impede dislocation motion. This may also be one of the reasons why the IC21 single-crystal alloy has excellent high-temperature stress-rupture strength.

2.3 Oxidation resistance

Figures 6a and 6b show the oxidation kinetic curves of IC21 single-crystal alloy after oxidation at 1100 and 1150 °C for 100 h, respectively. It can be seen that the alloy maintains a weight gain during cyclic oxidation; as the temperature increases, the oxidation rate of the alloy increases by a factor of 2. The oxidation kinetic curves at different temperatures obey a parabolic law, which is basically consistent with the oxidation characteristics of other single-crystal high-temperature alloys^[28,29]. The oxidation rates at the two temperatures are approximately 0.015 and 0.045 mg/(cm²·h), respectively. Both oxidation rates are relatively low, indicating that the alloy has excellent high-temperature oxidation resistance.

2.4 Casting process performance

To study the casting process performance of the alloy, this study designed and prepared complex thin-walled single-crystal castings as shown in Fig. 7a. The thicknesses of both the inner and outer walls of the double-layer wall are within the range of 0.5–1.0 mm. For different sections

Fig.4 Influence of temperature on yield strength and elongation of CMSX-4 and IC21 single crystal alloys^[21]

Table 2 Stress rupture properties of IC21 single crystal alloy at various load conditions^[13]

Temperature / °C	Orientation deviation / (°)	Stress / MPa	Life / h	δ / %
760	10	600	324.8	31.2
	4	750	69.8	29.5
	6		71.0	21.8
850	6	450	252.4	28.2
	7		271.8	27.4
	6	500	64.4	17.5
980	5		75.4	16.8
	8	22	121.9	50.2

Fig. 5

Figure 5: Fig. 5

Temperature / °C	Orientation deviation / (°)	Stress / MPa	Life / h	δ / %
1100	7	250	141.5	47.3
	8		76.5	43.7
	8		84.0	50.1
	10	140	170.5	12.1
	8	100	159.2	6.7
1150	8		110.0	10.8
	8		99.0	9.9

Fig. 5 Dislocation networks of IC21 single crystal alloy formed during high-temperature thermal exposure at 1100 °C for 1 h at low (a) and high (b) magnification, 10 h (c), and 100 h (d) ¹

Fig. 6 Cyclic oxidation mass-gain kinetic curves of IC21 single crystal alloy at 1100 °C (a) and 1150 °C (b)

Samples were taken at different positions for orientation and microstructural analysis; the results are shown in Fig. 7b-d, in order to examine the casting processability of the alloy under conditions of different wall thicknesses and different directional-solidification temperatures. It can be seen from the figure that the overall microstructure of the alloy contains no shrinkage porosity or inclusions. The results of X-ray Laue orientation testing show that the single-crystal integrity of the specimens is good; the crystal orientations at the three positions are consistent, and no low-angle grain boundaries are present. Measurements of the primary dendrite arm spacing indicate that the differences among the three positions are not large, with a size of approximately 500 μm . In Fig. 7b and c, at the 90° corner, the secondary dendrites in different positions of the two wall layers grow and coalesce along the primary dendrite trunk, with good continuity, forming a complete single crystal after solidification; moreover, no shrinkage porosity or inclusions are present at the corner, indicating that the IC21 alloy has good fluidity.

Fig. 7 Microstructure of complex thin-walled IC21 single crystal alloy (a) and enlarged views corresponding to areas A (b), B (c) and C (d) in Fig.7a

¹13,27

Fig. 6

Figure 6: Fig. 6

Fig. 7 Microstructure of complex thin-walled IC21 single-crystal alloy (a) and enlarged views corresponding to areas A (b), B (c), and C (d) in Fig. 7a

Figure 7: Fig. 7 Microstructure of complex thin-walled IC21 single-crystal alloy (a) and enlarged views corresponding to areas A (b), B (c), and C (d) in Fig. 7a

3 Conclusion

- (1) In view of the performance requirements for high-pressure turbine guide vanes, a low-density, low-cost Ni₃Al-based single-crystal alloy IC21 was developed, with Re content not greater than 1.5% and density below 8.0 g/cm³, and the heat-treatment schedule of the alloy was determined.
- (2) The incipient melting temperature of the IC21 single-crystal alloy is about 1345 °C. After solution heat treatment and aging heat treatment, the γ' phase is uniformly distributed, with a high degree of cuboidal morphology and ordering of arrangement; its size is about 420 nm and its volume fraction is about 80%. After long-term thermal exposure at 1080 °C, no topologically close-packed phase precipitates.
- (3) The tensile strength and yield strength of the IC21 single-crystal alloy at 1100 °C are 490 and 470 MPa, respectively. The stress-rupture life at 1100 °C and 140 MPa can reach 170.5 h, and that at 1150 °C and 100 MPa can reach 110.0 h.
- (4) The IC21 single-crystal alloy has good high-temperature oxidation resistance. The cyclic oxidation kinetic curves in air at 1100 and 1150 °C for 100 h follow a parabolic law, and the oxidation weight-gain rates are 0.015 and 0.045 mg/(cm² · h), respectively.
- (5) The IC21 alloy has good casting processability, and still exhibits good fluidity and single-crystal integrity within a thickness range of 0.5–1.0 mm.

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Note: Figure translations are in progress. See original paper for figures.

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