Postprint: Research on Irradiation Damage of Domestic Nuclear-Grade Stainless Steel

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Abstract

Irradiation experiments were conducted on domestically-produced nuclear-grade 304 stainless steel specimens using a 2 MeV proton beam at 360 °C. The irradiation damage of the material was investigated using a microhardness tester, transmission electron microscopy (TEM), and three-dimensional atom probe (3DAP), and the influence of irradiation dose on the evolution of irradiation damage was analyzed. The results indicate that the irradiation damage microstructure of 304 stainless steel is dominated by dislocation loops and a small number of voids, with the number density of dislocation loops on the order of 10^{22} m^{-3} and an average size of less than 10 nm. Elemental segregation occurs at grain boundaries and dislocation loops, where the segregation extent of Cr and Ni is similar at both locations, while the segregation extent of Si at dislocation loops is several times higher than that at grain boundaries. The average size and number density of dislocation loops, the degree of grain boundary segregation, and the extent of irradiation hardening all increase with increasing irradiation dose and tend to saturate within the 3-5 dpa range.

Full Text

Study of Irradiation Damage in Domestically Fabricated Nuclear Grade Stainless Steel

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Abstract

Radiation-induced segregation (RIS) and microstructural evolution, such as dislocation loops and cavities, are major microstructural causes for irradiation-assisted stress corrosion cracking (IASCC) of austenitic stainless steel (SS) core components. While several studies have reported on irradiation-induced damage in nuclear grade (NG) austenitic SS, the evolution of dislocation loop density and size and its correlation with mechanical properties remain incompletely understood. Additionally, the correlation between segregation at grain boundaries and that at dislocation loops has received limited attention. In particular, systematic studies of irradiation damage in domestically fabricated NG austenitic SS are still lacking.

In this work, proton-irradiation-induced microstructural damage in domestically fabricated 304NG SS was characterized to correlate RIS and dislocation loop density and size with irradiation dose, as well as to relate dislocation loop characteristics to radiation-induced hardening. The results revealed that radiation-induced microstructural damage consisted mainly of dislocation loops with a few micro-voids. The loop density was on the order of 10^{22} m ^{3} with an average size of <10 nm. The square root of the product of loop density and size scaled linearly with the square root of irradiation dose with a factor of 6.8×10^{3} (dpa) · /mm. The loops were believed to be primarily responsible for hardening in 304NG SS, which also scaled linearly with the square root of irradiation dose with a factor of 79.5 (dpa) · kg/mm^2. A comparative analysis of segregation at grain boundaries and dislocation loops showed that while the depletion of Cr and enrichment of Ni at dislocation loops and grain boundaries were similar, the enrichment of Si at dislocation loops could be about 6 times that at grain boundaries. Furthermore, loop density and size, as well as RIS and radiation-induced hardening, all increased with higher dose and tended to saturate at a dose of 3.0–5.0 dpa.

KEY WORDS: nuclear grade stainless steel, proton irradiation, dislocation loop, radiation-induced segregation, radiation-induced hardening

Introduction

Austenitic stainless steels are widely used as structural materials in light water reactor cores due to their excellent mechanical properties and corrosion resistance. Under the intense neutron irradiation and high-temperature/high-pressure water environment in the core, stainless steel materials undergo irradiation-assisted stress corrosion cracking (IASCC) during long-term service. Multiple IASCC failure events have occurred in stainless steel bolts used for baffle-former assembly in operating PWRs in Europe, America, and Japan. Irradiation-accelerated corrosion failures, represented by IASCC of core stainless steel materials and components, have become a critical issue affecting the safe and efficient operation of nuclear power plants.

Research has shown that irradiation-induced microstructural damage, radiation-induced segregation, and irradiation hardening produced by the interaction of intense neutron irradiation with stainless steel are key factors in IASCC occurrence. Irradiated materials experience irradiation hardening with increased yield strength, which promotes localized non-uniform deformation. The resulting narrow dislocation channels may induce IASCC. Cr depletion at grain boundaries caused by radiation-induced segregation reduces the corrosion resistance of grain boundaries and may promote intergranular stress corrosion cracking. Therefore, clarifying changes in microstructure, chemical composition, and mechanical properties induced by irradiation is a critical step in evaluating and predicting IASCC susceptibility.

Regarding irradiation-induced microstructural damage, research has clarified that it is primarily influenced by irradiation temperature, alloy composition, and irradiation dose. At irradiation temperatures exceeding 300 °C, irradiation-induced defects in austenitic stainless steels are mainly dislocation loops and voids. The size and number density of dislocation loops depend on alloy composition and irradiation dose. For example, significant dislocation loops and voids were produced in high-purity 304 stainless steel (HP 304) after proton irradiation at 360 °C, while only dislocation loops appeared in commercial-purity 304 stainless steel (CP 304) under the same conditions, with its size and number density differing by more than a factor of two from HP 304. Therefore, for different materials, the evolution of irradiation damage structure and dislocation loop size and density may vary significantly.

In radiation-induced segregation studies, a positive correlation between segregation and irradiation dose within a certain range has been established, influenced by irradiation dose and material factors (such as alloy composition, cold deformation, heat treatment, etc.). However, few studies have reported on solute atom segregation at dislocation loops, and whether it differs from segregation at grain boundaries remains unclear. Regarding irradiation hardening, it has been clarified that the essence of irradiation hardening is the pinning effect of numerous micro- and nano-scale defects such as dislocation loops and voids on dislocations, which hinders dislocation glide and causes material hardening.

Irradiation hardening is closely related to the size and number density of dislocation loops, but its quantitative relationship with mechanical properties remains controversial. For instance, the dispersed barrier hardening model suggests that irradiation hardening value (ΔH) is proportional to the square root of the product of dislocation loop number density (N) and diameter (d), while Edwards et al. argued that ΔH is not directly related to √(Nd).

The domestic production of nuclear materials and independent development of nuclear power technology are the foundation and guarantee for China' s nuclear power development. China has achieved domestic production of core structural materials, but research on irradiation-induced damage evolution is lacking, with extensive use of foreign data in material design and performance evaluation. This work selected domestically fabricated nuclear grade 304 stainless steel as the research object, used proton irradiation to simulate neutron irradiation, systematically analyzed the effects of irradiation on microstructure, elemental segregation, and mechanical properties, and quantitatively revealed the relationships between radiation-induced segregation, dislocation loop size, density and irradiation dose, as well as irradiation hardening and dislocation loop characteristics, providing performance validation data for domestic nuclear materials.

Experimental Methods

The experimental material was nuclear grade 304 stainless steel produced by a domestic nuclear material manufacturer, with chemical composition (mass fraction, %) of: C 0.04, Mn 1.73, Si 0.27, S 0.002, P 0.021, Ni 8.87, Cr 19.51, Co 0.04, Fe balance. Specimens measuring 20 mm × 3 mm × 2 mm were ground sequentially with water sandpaper to 3000 grit, then mechanically polished with 1.5 and 0.5 µm diamond paste, and finally manually polished with 40 nm SiO suspension for approximately 2 hours to ensure complete removal of the surface residual strain layer. Polished specimens were cleaned with deionized water and alcohol, then dried.

Irradiation experiments were performed at the Ion Beam Laboratory of the University of Michigan. A 2 MeV proton beam with 3 mm diameter was used to simulate neutron irradiation at 360 °C. The proton beam damage rate was approximately 6×10 dpa/s, with an effective irradiated region of 10 mm in the specimen center, as shown in [Figure 1: see original paper]. Under these irradiation conditions, the effective irradiation penetration depth at the specimen surface was approximately 20 µm. Further details of the irradiation process can be found in references [19,20].

After irradiation, microhardness measurements were first conducted on a Q-10A+ microhardness tester using a 25 g load to ensure the indenter penetration depth did not exceed the effective irradiation penetration depth, with a dwell time of 10 s. At least 30 valid measurements were obtained for each irradiation dose, with measurement positions shown as red lines in [Figure 1: see original paper]. The increase in hardness before and after irradiation was defined as ΔH.

Hardness-tested specimens were used to prepare TEM samples for observing irradiation-induced microstructural changes. First, specimens were electropolished for 5 s in a solution of 5% (volume fraction) perchloric acid and 95% methanol to remove approximately 3 μm of the irradiated surface layer. The electropolishing current was 30 mA at 30 °C. The opposite surface (non-irradiated side) was then ground sequentially with water sandpaper to 3000 grit and thinned to less than 60 μm thickness. Three 3 mm diameter disks were punched out, as shown in [Figure 1: see original paper]. Finally, the 3 mm disks were electropolished from the non-irradiated side using a TenuPol-5 thinner until electron transparency was achieved. The electrolyte was 10% perchloric acid and 90% alcohol at -30 °C with a voltage of 32 V.

Irradiation damage structures were observed using a JEM-2100 field emission TEM at 200 kV operating voltage. Bright-field (BF) and dark-field (DF) imaging were combined to observe dislocation loop morphology, with loop measurements and statistics taken at different specimen locations at similar magnifications. TEM specimen thickness at imaging locations was determined by electron energy loss spectroscopy (EELS). Dislocation loop number density was calculated as the total number of loops per unit area divided by the corresponding TEM specimen thickness [13,19]. Voids were observed and counted in slightly underfocused bright-field conditions [19]. Scanning transmission electron microscopy with energy-dispersive X-ray spectroscopy (STEM/EDS) was used to analyze elemental segregation at grain boundaries, with an EDS spot size of 0.5 nm and acquisition time of 20 s to avoid beam drift during analysis.

Intragranular chemical element segregation and atomic cluster distribution were characterized using three-dimensional atom probe (3DAP). Atom probe tips were prepared using an FEI QUANTA 200 3D focused ion beam (FIB) system, with the preparation procedure shown in [Figure 2: see original paper]. First, a Ga ion beam was used to cut a small rectangular block containing the irradiated region: a ~1 μm thick Pt layer was deposited on the specimen surface to mark the cutting position and protect the surface. The Pt-covered rectangular block was then cut out ([Figure 2b: see original paper]), the specimen stage was tilted by ~30°, and the block was welded to a specimen cantilever (W wire) with Pt. The connection between the block and specimen was severed, and the cantilever was moved to attach the block to a 3DAP tip base ([FIGURE:2c-f]). The deposited Pt on the surface and the sides of the rectangular block were removed by annular Ga ion beam sputtering, and finally thinned with a small current (0.1 nA) until an atom probe tip with curvature radius <50 nm was obtained ([FIGURE:2g-i]).

After preparation, specimens were analyzed in a CAMECA LEAP 4000X HR system using laser pulse mode with the following parameters: vacuum 7×10^{12} Pa, detection rate 1%, pulse rate 200 kHz, pulse energy 60 pJ.

2.1 Irradiation-Induced Microstructural Damage

[Figure 3: see original paper] shows bright-field TEM images of the microstructure in domestically fabricated nuclear grade 304 stainless steel before and after irradiation to various doses. Evidently, irradiation-induced microstructural changes including black dots and dislocation loops appeared, correlating strongly with irradiation dose. At low dose (0.5 dpa), black dot defect clusters began to appear with a few small dislocation loops ([Figure 3b: see original paper]). As dose increased to 1.5–5.0 dpa, black dots decreased significantly and eventually disappeared, with microstructural changes dominated by dislocation loops ([FIGURE:3c-e]). Dislocation loop size and number increased noticeably with irradiation dose, and loops did not overlap ([Figure 3f: see original paper]). [FIGURE:4a-e] shows dark-field images under g=200 (z=[110]) conditions, more clearly revealing changes in dislocation loop size and density with irradiation dose. [Figure 4f: see original paper] shows the corresponding diffraction pattern used for dark-field TEM observation.

Dislocation loop size distribution results are shown in [Figure 5: see original paper]. Loop diameters were mainly in the 2–15 nm range. At low doses (0.5 and 1.5 dpa), size distributions were relatively concentrated with high proportions of small loops, generally not exceeding 20 nm. At higher doses (3.0 and 5.0 dpa), size distributions became asymmetric with maximum sizes exceeding 30 nm. Quantitative analysis of average loop size and number density is shown in [Figure 6: see original paper]. Both loop size and density increased with irradiation dose. Loop size increased slowly with dose, stabilizing in the 3.0–5.0 dpa range, while loop density rapidly saturated in the 0–1.5 dpa range and remained on the order of 10^{22} m ³. At irradiation doses of 0.5, 1.5, 3.0, and 5.0 dpa, corresponding loop sizes were 5.81, 7.25, 8.61, and 9.34 nm, while loop densities were 0.364×10^{22}, 1.423×10^{22}, 1.804×10^{22}, and 2.014×10^{22} m ³, respectively ().

Void distribution in irradiated specimens was observed in under-focused bright-field TEM mode. As shown in [Figure 7: see original paper], void numbers were low in specimens irradiated to 0.5, 1.5, 3.0, and 5.0 dpa, with sizes in the 1–5 nm range showing no significant change with irradiation dose.

2.2.1 Grain Boundary Segregation

[FIGURE:8a-f] shows TEM/EDS analysis results at grain boundaries in unirradiated and irradiated specimens. No chemical segregation occurred at grain boundaries in unirradiated specimens, while irradiated specimens exhibited Ni and Si enrichment and Cr depletion at grain boundaries, with segregation regions of ~10 nm width and greater segregation near the boundary. [Figure 8g:

see original paper] shows grain boundary segregation as a function of irradiation dose. In the 0–3.0 dpa range, segregation increased with dose, gradually saturating at higher doses. At the maximum dose of 5.0 dpa, Cr depletion and Ni enrichment reached 5% (atomic fraction), while Si enrichment was approximately 5 times the matrix content.

2.2.2 Segregation at Dislocation Loops

[Figure 9: see original paper] shows 3DAP analysis of intragranular chemical distribution in a specimen irradiated to 3.0 dpa, revealing distinct Si-rich atomic clusters. To more intuitively characterize elemental segregation, isoconcentration surface analysis was performed. Since Si atoms readily segregate to defects such as dislocation loops and grain boundaries during irradiation, Si segregation can be used to reveal dislocation loop positions [21]. [Figure 10a: see original paper] shows a 2.5% (atomic fraction) Si isoconcentration surface plot. Compared with [Figure 9: see original paper], the matrix Si concentration was below this threshold and thus not displayed, clearly showing the distribution of irradiation-induced dislocation loops. By comparing isoconcentration surfaces of Si, Ni, Cr, and Mn at enriched Si locations, the overall elemental segregation at dislocation loops could be determined. [FIGURE:10b-e] shows isoconcentration surfaces at 2.5% Si, 16.0% Ni, 12.0% Cr, and 0.6% Mn, revealing Ni-Si clusters enriched in Ni and Si but depleted in Cr and Mn at dislocation loops.

A single dislocation loop was selected from [Figure 10a: see original paper] for quantitative segregation analysis. [Figure 11a: see original paper] shows elemental distribution at the dislocation loop, and [Figure 11b: see original paper] shows a 2D statistical profile along the radial direction (statistical region: 4 nm $\times$ 4 nm $\times$ 40 nm). Solute atoms segregated non-uniformly around the dislocation loop; for example, Ni, Si, Cr, and Mn contents at end A of the loop were approximately 30%, 70%, 15%, and 40% higher than at end B, respectively. Using isoconcentration surfaces of segregating solutes to identify cluster locations before selection is a convenient method for calculating cluster composition [22,23]. Based on all Ni-Si clusters identified by 0.8% Si isoconcentration surfaces, the average chemical composition of Ni-Si clusters was calculated to be 3.60% Si, 14.87% Cr, 12.18% Ni, and 69.35% Fe.

2.3 Irradiation Hardening

[Figure 12a: see original paper] shows microhardness at the irradiated surface as a function of irradiation dose. The material hardened significantly after irradiation, with hardness increasing with dose. The hardening rate slowed at doses of 3.0 and 5.0 dpa. [Figure 12b: see original paper] shows that irradiation hardening (ΔH) was proportional to the square root of irradiation dose with a proportionality coefficient of 79.5 (dpa) · kg/mm². At 5.0 dpa, ΔH increased to 171 HV, nearly three times that at 0.5 dpa ().

3.1 Irradiation-Induced Microstructural Damage

As shown in [Figure 3: see original paper], irradiation damage in domestically fabricated nuclear grade 304 stainless steel consisted mainly of dislocation loops with a few voids, consistent with literature results [13,24]. Bruemmer et al. [15] studied factors affecting irradiation damage in 300-series austenitic stainless steels, finding that microstructural damage is primarily influenced by irradiation temperature and dose. At temperatures exceeding 300 °C, irradiation-induced defects are dislocation loops 5–20 nm in size. Void formation requires higher temperatures, where enhanced interstitial diffusion promotes vacancy and bubble formation, which then aggregate into three-dimensional voids.

Dislocation loop number density is related to interstitial clustering during cascade collisions [18,25]. Proton irradiation creates cascade collisions in 304 stainless steel, forming vacancy-rich regions surrounded by interstitials in cascade zones. Vacancies collapse to form dislocation loops, a process also confirmed in xenon or other heavy ion-irradiated austenitic stainless steels [18]. Increased irradiation dose creates more and larger vacancy clusters, thus increasing loop size and density.

The observed evolution of loop size and density with dose was generally consistent with literature [15,25], but with significant differences in absolute values. As shown in [Figure 6: see original paper], loop density increased rapidly with dose, saturating at ~10^{22} m ³ by 1.5 dpa—about one order of magnitude lower than reported in [25]. Loop size increased more gradually, stabilizing at 3.0–5.0 dpa, while [25] reported size saturation at lower doses (~1.0 dpa). Loop size and density directly reflect irradiation-induced structural damage and can characterize damage degree. [Figure 13: see original paper] shows the square root of the product of loop density (N) and diameter (d) as a function of the square root of irradiation dose for both this study and [25]. For domestically fabricated 304 stainless steel, √(Nd) scaled linearly with √(dose) with a coefficient of 6.8×10^3 (dpa) · /mm, approximately half the literature value, indicating significantly less structural damage than reported for the same material in [25].

3.2 Irradiation-Induced Chemical Segregation

The elemental segregation at grain boundaries and dislocation loops shown in [Figure 8: see original paper] and [Figure 11: see original paper] results from unequal exchange rates between solute atoms and interstitials/vacancies [14,26]. Based on the inverse Kirkendall effect, the Perks model [27,28] provides a good explanation for solute segregation in irradiated materials:

[Equation would appear here based on original text]

where x represents solute atoms (Fe, Ni, Cr), D is the diffusion coefficient, is the jump distance, is the solute-vacancy exchange frequency, is the solute-interstitial exchange frequency, E is interstitial formation energy, K is Boltzmann constant, and T is irradiation temperature.

Using parameters from literature [29–32] and taking Cr diffusion as an example, > , meaning solute segregation in irradiated materials is primarily determined by . Therefore, only effects are discussed below.

Solute diffusion rate D is related to vacancy concentration C by [30]:

[Equation would appear here]

Based on literature parameters [29–32], the diffusion rate ratios for Fe, Ni, and Cr atoms were calculated. Radiation-induced segregation involves solute atoms migrating to grain boundaries with vacancies (schematic in [Figure 14: see original paper]), essentially an atomic flux. Segregation degree can be expressed as a function of solute concentration and diffusion rate. Since the sum of Fe, Ni, and Cr segregation equals 100%, normalization yields Cr depletion determined by the ratio of diffusion rates [30]:

[Equation would appear here]

where J_Cr is Cr atom flux and J_Fe is Fe atom flux. The diffusion rates follow D_Fe > D_Ni > D_Cr, meaning Cr diffuses faster than Ni, resulting in Ni enrichment and Cr depletion at grain boundaries in irradiated 304 stainless steel ([Figure 8: see original paper]). The degree of Cr depletion and Ni enrichment depends on both the interaction strength of Cr and Ni with point defects and alloy composition. With increasing irradiation dose, defect size and number increase (C in Eq. (3)), enhancing solute diffusion rates, thus explaining the observed increase in grain boundary segregation with dose ([Figure 8g: see original paper]). Fe segregation at grain boundaries depends on its relative diffusion coefficient to other solutes and the relative Cr and Ni contents. Allen et al. [33] observed Fe depletion at grain boundaries in most austenitic stainless steels, but Fe enrichment can occur when the Ni/Cr ratio is low such that Ni enrichment is less than Cr depletion.

[Figure 15: see original paper] shows compositional segregation analysis at grain boundaries and dislocation loops after 3.0 dpa irradiation. Overall, segregation at dislocation loops was similar to that at grain boundaries, showing Fe and Cr depletion with Si and Ni enrichment, and Fe, Cr, and Ni segregation levels were comparable at both features. However, Si segregation at dislocation loops was several times higher than at grain boundaries. Jiao et al. [21] confirmed this phenomenon in Si-added 304 stainless steel, which may be related to Si diffusion mechanisms. During segregation, Si undergoes uphill diffusion via interaction with interstitials, while Fe, Cr, and Ni primarily interact with vacancies [15]. The non-uniform segregation at dislocation loops ([Figure 10: see original paper] and [Figure 11: see original paper]) suggests that new phases may form when segregation reaches certain composition thresholds. Indeed, researchers [34,35] have observed G-phase precipitation at dislocation loops in high-dose irradiated stainless steels, with Si/(Si+Ni) content of ~25.0%, higher than the 22.8% in Si-Ni clusters. Therefore, the observed Si-Ni clusters may be G-phase precursors that could evolve into G-phase with continued irradiation and further Si, Ni segregation at dislocation loops.

3.3 Irradiation-Induced Mechanical Property Changes

The generation of numerous micro- and nano-scale defects that impede dislocation motion is the cause of irradiation hardening [16,17,36,37]. The dispersed barrier hardening (DBH) [14,16] and source hardening [17] models can well explain the relationship between microstructural changes and irradiation hardening. In the DBH model, irradiation hardening ΔH is proportional to the square root of the product of dislocation loop density and diameter:

[Equation would appear here]

[Figure 16: see original paper] shows fitting curves of ΔH versus √(Nd) based on this study and literature [25], yielding a proportionality coefficient between irradiation hardening ΔH (kg/mm^2) and √(Nd) (mm^{1}) for domestically fabricated 304 stainless steel of 11.6×10^{3} kg/mm. Since loop size and density are positively correlated with irradiation dose ([Figure 6: see original paper]), √(Nd) also increases with dose ([Figure 12: see original paper]). Studies [37,38] indicate that modulus mismatch between irradiation-formed atomic clusters and the matrix can also cause hardening.

The source hardening model proposes that yield strength increase can be characterized by loop density and size parameters:

[Equation would appear here]

where Δ is yield strength increment, M=3.06 is the Taylor coefficient for fcc structures, =0.6 is the loop strengthening factor, =76 GPa is the shear modulus, and b=2.5×10 cm is the Burgers vector magnitude. From Eqs. (7) and (8), the relationship between Δ and ΔH for domestically fabricated 304 stainless steel is:

[Equation would appear here]

Higgy et al. [39] statistically derived an empirical relationship for irradiated 304 stainless steel:

[Equation would appear here]

Clearly, Δ calculated from Eq. (9) and Eq. (10) shows significant differences. Using empirical Eq. (10) to predict irradiation hardening of domestic nuclear grade stainless steel would deviate from actual conditions, primarily due to material factors. The empirical equation considers effects of dislocation loops, vacancies, and voids, while few voids were observed in this experiment with very low density and size.

Conclusions

1. Irradiation damage in 304 stainless steel consisted mainly of dislocation loops and a few voids, accompanied by radiation-induced segregation and hardening. Average loop size and density, grain boundary segregation degree, and irradiation hardening all increased with irradiation dose and tended to saturate in the 3.0–5.0 dpa range.
2. Dislocation loop density in irradiated 304 stainless steel stabilized at 10^{22} m ^{3} with average size <10 nm. The square root of the product of loop density and diameter, √(Nd), was proportional to the square root of irradiation dose with a coefficient of 6.8×10^{3} (dpa) ・ /mm. Void sizes were generally <5 nm and showed no significant change with irradiation dose.
3. Irradiation caused Ni and Si enrichment and Fe and Cr depletion at both grain boundaries and dislocation loops in 304 stainless steel. Ni, Cr, and Fe segregation levels were similar at both features, while Si segregation at dislocation loops was several times higher than at grain boundaries.
4. Post-irradiation ΔH in 304 stainless steel was proportional to the square root of irradiation dose with a coefficient of 79.5 (dpa) ・ kg/mm^{2}, and ΔH was also proportional to √(Nd) with a coefficient of 11.6×10 ^{3} kg/mm.
5. The quantitative relationships between dislocation loop size/density and irradiation dose, and between irradiation hardening and loop characteristics, differed from literature values. The √(Nd) versus √(dose) coefficient was about half the literature value, while the ΔH versus √(Nd) coefficient was 1.7 times the literature value.

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