Grain boundary engineering for enhancing intergranular damage resistance of ferritic/martensitic steel P92

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Abstract

Ferritic/martensitic (F/M) steels have been widely used as structural material for thermal and nuclear power plants. However, it is susceptible to intergranular damage in service conditions, which is expected to be critical issues. In order to improve the intergranular damage resistance of F/M steel, thermomechanical process (TMP) was employed to achieve a grain boundary engineering (GBE) microstructure in a kind of F/M steel P92 in this study. TMP, including cold rolling to 6%, 9%, and 12% thickness reduction, respectively, followed by austenization at 1323 K for 40 min plus tempering at 1053 K for 45 min, were applied on as-received (AR) P92 steel. Both prior austenite grain (PAG) size, prior austenite grain boundary character distribution (GBCD), and the connectivity of prior austenite grain boundaries (PAGBs) were investigated. Compared with AR specimen, the PAG size does not change significantly. The fraction of coincident site lattice boundaries (CSLBs, $3 \leq \Sigma \leq 29$) and Σ3n boundaries along PAGBs decreases with increasing reduction ratio due to the recrystallization fraction increases with increasing reduction ratio. The PAGBs connectivity of the 6%-deformed specimen deteriorates compared with that of AR specimen slightly. Moreover, the potentiodynamic polarization studies revealed that the intergranular damage resistance of studied steel could be improved by enhancing the fraction of CSLBs along PAGBs, indicating that TMP, involving low deformation, could enhance the intergranular damage resistance.

Full Text

Grain Boundary Engineering of Ferritic/Martensitic Steel P92 for Enhancing Intergranular Damage Resistance

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ABSTRACT

Ferritic/martensitic (F/M) steel is widely used as a structural material in thermal and nuclear power plants; however, it is susceptible to intergranular damage, which represents a critical issue under service conditions. In this study, a thermomechanical process (TMP) was employed to achieve a grain boundary engineering (GBE) microstructure in F/M steel P92 to improve its resistance to intergranular damage. The TMP, which included cold-rolling thickness reductions of 6%, 9%, and 12%, followed by austenitization at 1323 K for 40 min and tempering at 1053 K for 45 min, was applied to as-received (AR) P92 steel. The prior austenite grain (PAG) size, prior austenite grain boundary character distribution (GBCD), and connectivity of prior austenite grain boundaries (PAGBs) were investigated. Compared to the AR specimen, the PAG size did not change significantly. The fraction of coincident site lattice boundaries (CSLBs, $3 \leq \Sigma \leq 29$) and $\Sigma 3n$ boundaries along PAGBs decreased with increasing reduction ratio because the recrystallization fraction increased with the reduction ratio. The PAGB connectivity of the 6% deformed specimen slightly deteriorated compared with that of the AR specimen. Moreover, potentiodynamic polarization studies revealed that the intergranular damage resistance of the studied steel could be improved by increasing the fraction of CSLBs along the PAGBs, indicating that the TMP involving low deformation could enhance the intergranular

damage resistance.

Keywords: Grain boundary engineering, Ferritic/martensitic steel, Prior austenite grain boundary character distribution, Grain boundary connectivity, Intergranular damage resistance

1. INTRODUCTION

Ferritic/martensitic (F/M) steels have been widely used as structural materials in thermal and nuclear power plants and are candidate fuel cladding materials in advanced Gen-IV reactors because they exhibit higher thermal conductivity, lower cost, and greater resistance to radiation-induced void swelling than austenitic stainless steels. As an advanced F/M steel, P92 steel is microalloyed with vanadium and niobium, and its boron and nitrogen contents are carefully controlled. Owing to its superior high-temperature strength and better creep properties compared to other F/M steels, P92 steel has been used or considered for preferential application in critical thermal and nuclear power plant components such as main steam pipes, reheat steam pipes, boilers, and turbines. However, relevant studies have shown that high-temperature steam environments and salt accumulation can accelerate the corrosion of steam pipelines and even cause catastrophic pipe bursts. Therefore, improving the intergranular corrosion resistance of P92 steel is of great significance.

Previous studies have revealed that sensitization—specifically, chromium depletion resulting from chromium carbide precipitation at grain boundaries (GBs)—leads to degradation of intergranular corrosion resistance. Zhou et al. and Randle et al. reported that special GBs, namely coincident site lattice boundaries (CSLBs), strongly suppress chromium carbide precipitation. Consequently, grain boundary engineering (GBE) is believed to be an economical and effective solution for improving resistance to chromium depletion and suppressing carbide precipitate formation along GBs by introducing CSLBs. In F/M steels, prior austenite grain boundaries (PAGBs) serve as preferential sites for intergranular corrosion. Thus, previous studies have demonstrated that increasing the fraction of CSLBs ($3 \leq \Sigma \leq 29$) along PAGBs can enhance the intergranular corrosion resistance of F/M steel. GBE involves a series of thermomechanical processes (TMPs) that can improve the grain boundary character distribution (GBCD) and disrupt random boundaries without disturbing the original microstructure by increasing the fraction of CSLBs along PAGBs. In recent decades, GBE has been successfully applied to several nickel-based alloys and austenitic steels. Building on these successful applications, researchers have attempted to introduce the GBE method for modifying F/M steel. To the best of our knowledge, a TMP can effectively achieve a GBE microstructure in metallic materials with low stacking fault energy (SFE). However, few studies have used TMP to investigate the prior austenite GBCD in F/M steel because materials with bcc structures have higher SFE than those with fcc structures, and the mechanism of GB evolution and transformation in F/M steel is not well understood and requires systematic analysis. Therefore, it was necessary to apply the GBE method to study the effects of different TMPs on PAGBs.

Electrochemical corrosion testing is an important method for investigating the corrosion resistance of materials and has been shown to be environmentally friendly, economical, nondestructive, quantitative, and useful for corrosion studies in laboratory investigations. Over the last decade, significant research on the corrosion behavior of 304 stainless steel, rusted steel, EH47 ship steel, and titanium-clad carbon steel has been successfully conducted using electrochemical corrosion tests. Thus, electrochemical techniques can provide a reliable basis for evaluating the corrosion resistance of the studied specimens.

The purpose of this study was to evaluate the effects of different deformation levels on P92 steel. Prior austenite grain (PAG) size, prior austenite GBCD, PAGB connectivity, and carbide particles were investigated via metallographic analysis and scanning electron microscopy (SEM)/electron backscatter diffraction (EBSD). Corrosion tests were conducted to verify the feasibility of improving corrosion resistance. The corrosion performance of P92 steel in 0.03 M Na_2SO_4 and 0.03 M NaCl aqueous solutions was determined through potentiodynamic polarization tests. The results are expected to provide insights into methods for enhancing the intergranular damage resistance of P92 steel.

2.1 Experimental Material

A commercial F/M steel P92 pipe with a wall thickness of 60 mm in the as-fabricated condition was selected as the initial material for this study, and its mechanical properties have been reported in the literature. The pipe underwent a final heat treatment consisting of normalizing and tempering (NT). Normalizing was conducted at 1323 K for 30 min, while tempering was performed at 1038 K for 60 min, after which the pipe was cooled to room temperature. The chemical composition of the pipe was 0.093C, 0.14Si, 0.41Mn, 8.75Cr, 1.62W, 0.183V, 0.052Nb, 0.063N, 0.207Ni, 0.505Mo, 0.001Ti, 0.003B (wt%), with the balance being Fe. Previous studies have suggested that strain-induced boundary migration (SIBM) can improve the fraction and distribution of CSLBs along PAGBs. Consequently, steel plates with dimensions of 300 mm × 60 mm × 2.5 mm were subjected to a TMP involving three cold-rolling reduction ratios—namely, 6%, 9%, and 12%—followed by austenitization at 1323 K for 40 min, tempering at 1053 K for 45 min, and air cooling. [Figure 1: see original paper] shows the TMP routes for all specimens. The as-received and 6%, 9%, and 12% deformed specimens are designated as AR, 6D, 9D, and 12D, respectively. The nomenclature of each specimen is listed in . The specimens were cut normal to the rolling direction to observe the microstructure, with the observation area located at the edge of the specimen.

2.2 Microstructural Investigation

Specimens for EBSD analysis with dimensions of 10 mm × 10 mm × (2.2–2.35) mm were cut from the interior of the TMP-treated steel plates, and the EBSD scanning area was oriented vertical to the plate surface. The specimens were first mechanically ground with SiC paper of various grits up to 5000 grit, then mechanically polished with diamond solution of different grain sizes up to 1 m. Final polishing was performed with 0.04 m silica solution. The GBs were quantitatively evaluated using a ZEISS Gemini SEM 500 scanning electron microscope equipped with a Nordlys Max3 EBSD probe. The acceleration voltage was 20 kV, the beam current was 10 A, and the scanning step size was 0.09–0.2 m. This work adopted Brandon's criterion to categorize CSLBs: $\Delta \ \leq 15^\circ\Sigma^{-1}/^{2}$, and boundaries with $2^\circ \leq \ < 10^\circ$ were considered low-angle grain boundaries (LAGBs). After EBSD scanning, Aztec Crystal 2.1 software was used to reconstruct the PAGs, assuming a Kurdjumov–Sachs (K–S) orientation relationship. Aztec Crystal 2.1 software was also used for subsequent analyses.

The EBSD maps of the AR specimens are shown in [Figure 2: see original paper]. The maps were cropped to 100 m × 50 m for better visualization of the microstructure. A typical lath-martensite microstructure is evident in the figure. The inverse pole figure (IPF) maps of the AR specimen after PAG reconstruction clearly show that the martensite laths disappear and PAGBs become visible during the reconstruction process. The reconstructed PAG size is approximately 20 m, which is similar to the metallographic result and the value obtained by T. Sakthivel et al. Relevant statistical results will be analyzed in the following sections.

Compared with the EBSD technique, metallographic analysis can observe a larger area, which ensures greater accuracy in PAG size measurement. In the metallographic method used to reveal the microstructure, the specimens were ground, polished, and etched in a solution consisting of 1.5 mL nitric acid (superior grade), 1 mL hydrofluoric acid (analytical grade), 2 mL hydrogen peroxide (30%, analytical grade), 10 mL detergent (containing surfactant), and 50 mL distilled water for 2–10 s. The linear intercept method was used to measure PAG size from the metallographic images. All images contained more than 500 PAGs, and more than 30 lines were randomly drawn on each image.

2.3 Corrosion Test

Electrochemical corrosion tests were performed to study the effect of TMP on intergranular damage resistance. For these tests, specimens were wire-cut to dimensions of 10 mm × 2 mm × (2.2–2.35) mm. Each electrochemical specimen was welded to a copper conductor and sealed with epoxy resin, resulting in an exposed working surface area of 0.22–0.235 cm^2. The surfaces were polished with sandpaper and wiped with anhydrous alcohol. The Tafel curve was measured in 0.03 M Na_2SO_4 and 0.03 M NaCl aqueous solution at room temperature (RT) using a three-electrode system and CHI760e electrochemical workstation, consistent with the experimental environment of Pang et al. The working electrode was the specimen, the reference electrode was a saturated calomel electrode (SCE), and the auxiliary electrode was a platinum sheet. The experiment commenced once the steady-state open-circuit potential (OCP) was reached, and the Tafel curve was obtained over a scanning range of −1 to 1 V with respect to the OCP at a scanning rate of 0.005 V/s. To ensure reproducibility, all corrosion experiments were repeated at least three times.

3.1 Prior Austenite Grain Size

The grain structures of TMP-treated P92 steel with different reduction ratios are shown in [Figure 3: see original paper]. For better visualization, the images were cropped to 250 m × 220 m. The distribution of PAGBs and martensitic lath boundaries is evident in the figure, indicating the typical microstructure of P92 steel.

Considering the specimen thickness and low cold-rolling reduction ratios, the PAG size was measured in two areas as shown in Figure 4: see original paper: one just beneath the surface, designated as the ‘edge’ area, and the other at the specimen center, designated as the ‘middle’ area. Based on metallographic analysis, the PAG sizes of the AR and TMP-treated specimens as a function of reduction ratio are presented in Figure 4: see original paper. Compared with the AR specimen, the PAG size of all TMP-treated specimens did not change significantly, which is consistent with the results reported in the literature. However, the PAG sizes of specimens with different reduction ratios exhibited slight variations. The PAG size in the edge area decreased with increasing reduction ratio, whereas the PAG size in the middle area increased with increasing cold-rolling reduction ratio up to 9% and then decreased at a reduction ratio of 12%. The literature shows that PAG growth dynamics are directly related to the degree of deformation, temperature, and time. Under isothermal growth conditions, when the temperature exceeds the PAG growth temperature Ac_1, higher peak temperatures and longer holding times produce larger PAG sizes under ideal conditions. In this study, the normalizing temperature and time were identical, and the tempering temperature was lower than the PAG growth temperature. Therefore, the heat treatment process should have had the same effect on PAG size. In conclusion, the strain introduced by cold rolling was the only factor affecting the PAG size of the TMP-treated specimens. The recrystallization fraction, determined by the grain orientation spread (GOS) value, was positively correlated with the reduction ratio in this study, as shown in Figure 4: see original paper, indicating that the strain introduced by cold rolling promotes recrystallization. Only grains with $0° < \mathrm{GOS} < 1°$ were identified as recrystallized grains, and this criterion was consistent with the experimental results. SIBM promotes grain growth, whereas recrystallization reduces grain size. The relationship between PAG size and recrystallization fraction calculated from the EBSD data in the edge area is consistent with this discussion. However, this appears to contradict the changing trend of PAG size in the middle area. The following provides a detailed explanation for this contradiction. As shown in Figure 4: see original paper, the edge and middle areas of 12Cr F/M steel also exhibit different trends in PAG size when the annealing time is 45 min. Kinoshita et al. noted that this duration did not appear sufficient for grain growth in the middle area at low reduction ratios. In our work, the variation trend of PAG size in the middle area with reduction ratio was inconsistent with that of the recrystallization fraction because we collected EBSD data from the edge area.

Based on the above analysis, we can conclude that SIBM dominates at low strains, while recrystallization dominates as the strain increases. GB migration is usually accompanied by twin formation, and recrystallization typically introduces new random boundaries. Therefore, we can expect that a high fraction of twin boundaries, corresponding to the {111} Σ3 boundaries, will be introduced into the austenite phase for the 6D specimen, and the fraction of CSLBs along PAGBs will decrease with increasing reduction ratio, which will be discussed in the next section.

3.2 Prior Austenite Grain Boundary Character Distribution

The EBSD maps of the AR and TMP-treated specimens after PAG reconstruction are shown in Figure 5: see original paper. Different types of PAGBs are marked with colored line segments. For better visualization, the maps were cropped to 100 m × 50 m. Compared with the AR specimen shown in Figure 5: see original paper, Σ3n boundaries still account for the majority of CSLBs along the PAGBs after TMP. To ensure statistical accuracy, at least 500 PAGBs were sampled for analysis.

The fractions of CSLBs along the PAGBs of the studied steels are presented in Figure 5: see original paper. The TMP clearly led to higher fractions of CSLBs along PAGBs, with a high fraction of Σ3 boundaries observed along PAGBs. Compared with the AR specimen, the fraction distribution of Σ3 boundaries along PAGBs for 6D, 9D, and 12D improved by 46.3%, 27.1%, and 8.3%, respectively. Figure 6: see original paper shows the fraction of Σ3n boundaries along PAGBs as a function of reduction ratio for the AR and TMP-treated specimens. The fraction of Σ3n boundaries along PAGBs was evaluated as follows: $(\gamma) =$ f(Σ3n) L(Σ3n) / L(total), where L(Σ3n) is the total length of Σ3n boundaries along PAGBs and L(total) is the total length of PAGBs. The variation trend of the Σ3n boundary fraction along PAGBs with reduction ratio is similar to that of PAG size, decreasing with increasing reduction ratio. Figure 6: see original paper shows the relationship between the fraction of CSLBs along PAGBs and the reduction ratio. The fraction of CSLBs along PAGBs also decreased with increasing reduction ratio. This finding is consistent with the results of Shimada et al. and Kokawa et al., who found a peak in the CSLB fraction at 5% deformation and an inverse relationship between the CSLB fraction and reduction ratio when deformation exceeded 5% in γ-Fe. For comparison, data obtained from 10Cr and 12Cr F/M steels are shown in Figure 6: see original paper, and data from 9Cr and 10Cr F/M steels are shown in Figure 6: see original paper. As mentioned in Section 3.1, SIBM can promote twin formation, whereas recrystallization can introduce new random boundaries. However, the mechanism dominating at a given deformation level remains unclear. We propose a critical deformation value, σ, for different materials and TMPs. When the deformation is less than σ, SIBM is dominant; otherwise, recrystallization dominates instead of SIBM. In practice, σ differs for different materials and TMPs. As can be observed from [Figure 6: see original paper], σ is 10% in the work of Hirayama et al. (10Cr F/M steel) and 5% in the work of Kinoshita et al. (12Cr F/M steel). It can be reasonably inferred that the deformation exceeded σ in this work, which is confirmed by the recrystallization fraction discussed in Section 3.1 and the observed trends in Σ3n and CSLB fractions along PAGBs.

3.3 Prior Austenite Grain Boundary Connectivity

[Figure 7: see original paper] shows the two-dimensional PAGB network for the four steels, where green and black lines denote special boundaries (Σ3–Σ29b boundaries) and random boundaries, respectively. In addition to the different fractions of Σ3n boundaries and CSLBs along PAGBs, the distribution of special boundaries and the degree of connectivity vary significantly among the four studied steels. With the application of the cold-rolling–normalizing–tempering process, the random prior austenite GBs were segmented into fragments.

Percolation theory is typically used to simulate GB connectivity. Previous studies have suggested that four types of triple junctions can occur in materials: TJ0 (R-R-R), TJ1 (S-R-R), TJ2 (S-S-R), and TJ3 (S-S-S), as shown in Figure 8: see original paper. To assess GB connectivity, it is necessary to consider the distribution of triple junctions. Any triple junction containing at least one random boundary is susceptible to corrosive attack. However, no TJ2 or TJ3 triple junctions were observed in any of the specimens, as shown in Figure 8: see original paper. Only the 6D specimen exhibited more TJ1 and fewer TJ0 triple junctions than the AR specimen. The triple junction distributions of the other specimens were almost identical to those of the AR specimen. This indicates that only the 6D specimen had more fragmented prior austenite random boundaries than the AR specimen, whereas the other specimens showed little change in prior austenite random boundary interruption compared to the AR specimen.

3.4 Carbide Particles

The SEM micrographs in [Figure 9: see original paper] show the distribution of precipitates in the AR, 6D, 9D, and 12D specimens. One of the authors has demonstrated that Cr-rich $M_{23}C_6$ is the main precipitate phase in P92 steel when the tempering temperature exceeds 700 °C. Subblock boundaries and PAGBs act as preferential nucleation sites for Cr-rich $M_{23}C_6$, and GBE can enhance the fraction of subblock boundaries, leading to finely dispersed precipitates in the studied steel. Furthermore, CSLBs along PAGBs can strongly suppress the formation and coarsening of chromium carbide precipitates, as mentioned in the Introduction, because the diffusivity of CSLBs is generally lower than that of random boundaries. A quantitative analysis of the precipitate phase requires further investigation, which is beyond the scope of this study. However, based on the above analysis, it can be inferred that the 6D, 9D, and 12D specimens have more densely dispersed precipitates than the AR specimen, as shown in [Figure 9: see original paper], which is consistent with results reported by Hirayama et al. and Gupta et al. This suggests that GBE-treated steel can achieve better high-temperature properties than AR specimens. Experimental measurements of high-temperature properties will be conducted in future studies. Meanwhile, it can also be inferred that corrosion resistance will be improved in GBE-treated materials as chromium segregation is suppressed due to the enhanced fraction of CSLBs along PAGBs, as discussed in Section 3.2.

3.5 Corrosion Behavior

Increasing the fraction of CSLBs along PAGBs and disrupting prior austenite random boundaries are beneficial for improving resistance to intergranular damage. Based on the analyses in Sections 3.1, 3.2, and 3.3, a TMP with a low reduction ratio can enhance the fraction of CSLBs along PAGBs and disrupt prior austenite random boundaries, thereby improving intergranular resistance. In this study, electrochemical corrosion tests were conducted to determine whether these changes in prior austenite GBCD and PAGB connectivity are associated with corrosion resistance. All corrosion tests were repeated at least three times, and representative data are presented.

Potentiodynamic polarization curves were measured to determine the self-corrosion current density (i_{corr}) and self-corrosion rate (v), as shown in Figure 10: see original paper, and to verify the effectiveness of GBE. The i_{corr} values were obtained by extrapolating the potentiodynamic polarization curves, and v was calculated according to the following formula: i_{corr}, where M is the molecular mass of the material, n is the number of transferred electrons, and F is Faraday's constant. The corresponding data are listed in .

The variations of i_{corr} and corrosion rate v followed a trend opposite to that of the CSLB fraction along PAGBs, as shown in Figure 10: see original paper. As the fraction of CSLBs along PAGBs increased, the i_{corr} values were $(1.02 \pm 0.15) \times 10^{-5}$, $(1.53 \pm 0.41) \times 10^{-5}$, $(3.16 \pm 0.19) \times 10^{-5}$, and $(3.57 \pm 0.25) \times 10^{-5}$ $\mathrm{A \cdot cm^{-2}}$, respectively, and the v values were 3.12 ± 0.46, 4.66 ± 1.25, 9.66 ± 0.58, and 10.92 ± 0.74 $\mathrm{mil \cdot year^{-1}}$, respectively. The self-corrosion current density and rate of the TMP-treated specimens were lower than those of the AR specimen, reflecting improved electrochemical corrosion resistance. The specimen with a higher fraction of CSLBs along PAGBs showed decreased electrochemical corrosion susceptibility, proving that GBE is effective in improving the intergranular damage resistance of P92 steel under the presented conditions. This result is also consistent with visual inspection of the metallographic analysis.

Typical SEM micrographs of AR and TMP-treated specimens after potentiodynamic polarization testing in 0.03 M Na_2SO_4 and 0.03 M NaCl aqueous solutions are shown in Figure 11: see original paper. The electrochemical corrosion morphologies of the AR specimens exhibit larger and denser corrosion pits than those of the TMP-treated specimens. Additionally, high-magnification micrographs of the AR specimen reveal the generation of corrosion cracks, as shown in Figure 11: see original paper, which were not observed in the other specimens, as shown in Figure 11: see original paper. The electrochemical corrosion morphologies of the 9D and 12D specimens show fewer corrosion pits than those of the AR specimen, as shown in Figure 11: see original paper, and their surfaces are smoother than those of the AR specimen. The electrochemical corrosion morphologies of the 6D specimen exhibited the lowest number of corrosion pits and demonstrated the least degree of corrosion compared with the other specimens, as shown in Figure 11: see original paper.

4. CONCLUSION

Through metallographic analysis and EBSD testing, the PAG size, prior austenite GBCD, and prior austenite grain boundary connectivity of F/M steel P92 under different TMPs were investigated. Electrochemical corrosion tests were performed to verify the validity of GBE. The major findings of this study are as follows:

The PAG size of all TMP-treated specimens did not change significantly compared with that of the AR specimen. The PAG size decreased with increasing reduction ratio in the edge area due to the increasing recrystallization fraction. The PAG size increased with deformation up to 9% and then decreased in the middle area because the normalizing time was relatively short.

Different critical deformation values, σ, were found for different materials and TMPs. When the deformation was lower than σ, SIBM was dominant; otherwise, recrystallization became dominant. In this work, the fraction of CSLBs along PAGBs decreased with increasing reduction ratio because the deformation exceeded σ.

Potentiodynamic polarization studies revealed that the electrochemical corrosion resistance of P92 steel can be improved by introducing a high fraction of CSLBs along PAGBs under the presented conditions. Therefore, the GBE method is feasible and effective for improving the intergranular damage resistance of F/M steels.

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AUTHOR CONTRIBUTIONS

All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Lei Peng, Shang-Ming Chen, Jing-Yi Shi, Yin-Zhong Shen, and Hui-Juan Wang. The first draft of the manuscript was written by Lei Peng, Shang-Ming Chen, and Jing-Yi Shi, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are openly available in Science Data Bank at https://cstr.cn/31253.11.sciencedb.j00186.00379 and https://www.doi.org/10.57760/sciencedb.j00186.00379.

CONFLICT OF INTEREST

The authors declare that they have no competing interests.

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