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Dislocation Crack Interactions and Lattice Rotation Under Uniaxial and Cyclic Loading

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We investigate here th accumulation of fatigue of We report a significant lat X-ray diffraction (XRD) crack tip showed variatio that of the 200 peak as we of large number of disloc cyclic deformation cause stress cannot be explained is a major factor controlli- suggests a new the energy	e configuration o lamage, which is tice rotation in th and transmission ns in the relative e approach the cra ations along {111 s 200% increase i l by the difference ing the stress. The y dissipation mec	f dislocations emitted b still unknown and is cru e plastic zone ahead of a electron microscopy (T intensities of 111, 200 an ck, suggesting lattice ro } planes across grain bo n residual stress in Al 70 e in dislocation density a ese findings provide new hanism with implication	y a sharp crack tip in stu- icial for understanding t a fatigue crack at room to 'EM). A series of high r nd 220 Al peaks. The int tation as a result of fatig bundaries, which results 1075, but decrease in resi- lone in the plastic zone. v insights on the role of as for predicting materia	ructural alloys, and he underlying phys emperature in Al 11 esolution XRD sca tensity of 111 peaks gue crack growth at in increase in the r dual stress by 80% We demonstrate tha lattice rotation on t il life under cyclic l	the characteristics of plastic zone as a result of the ics of the deformation process ahead of a crack tip. 00 (H14) and Al 7075 (T7), observed by employing ns measured at different locations in the vicinity of was observed to gradually increase as compared to room temperature. We ascribe such rotation to glide misorientation during crack growth. In addition, the in commercially pure Al. Such a change in residual t the deformation associated with the lattice rotation he deformation process under fatigue loading. This oad.
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Dislocation Crack Interactions and Lattice Rotation Under Uniaxial and Cyclic Loading

We present here the closeout report on our project, *3-D Electron tomography of dislocation processes and ductility loss phenomena*. The project was aimed at enhancing our understanding of fracture and failure of metallic materials via investigation of the configuration of dislocations emitted by a sharp crack tip in structural alloys. A significant outcome of this work as described below has been the development of a new mechanism for crack tip deformation behavior under unidirectional and cyclic loadings. We demonstrated that the cyclic deformation behavior in Al alloys is associated with the changes in dislocation configurations, lattice rotation and increase in residual stress. In addition, this core program has generated a number peer reviewed publications and presentations. We have provided a list of publications (11 papers including two book chapters), presentations (15 presentations including invited and contributed) and a patent generated from this program.

Despite the many years of fracture and fatigue research, there is still an incomplete understanding of crack formation and growth. An improved understanding of crack tip deformation is a key component of managing crack growth and failure. Ultimately research in crack tip behavior leads to an improved understanding of structural failure in metals and alloys. If we understand the crack cycle and associated failure better, we can predict failure more accurately which in turns allows for improvements in removal for cause, maintenance planning and life cycle management of the component and its platform. These factors lead to better control on maintenance costs (planned maintenance is typically much less expensive than unplanned maintenance), avoidance of catastrophic failure such seen recently on commercial airlines, readiness and logistics planning. All of these factors are critical to maintaining the operational capability of the Navy.

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We were able to identify, in the course of this program on 3D dislocation tomography, that a previously unresolved key issue has been the relationship between the plastic zone size and the number of dislocations emitted from a crack tip. Significant efforts have been made in attempts to understand the crack-tip deformation behavior in metals and alloys during tensile deformation in an electron microscope. It is found that deformation occurs mostly by the emission of dislocations from the crack tip, which acts as a possible dislocation source because it is associated with the highest stress. The behavior of dislocations ahead of the crack tip has been correlated with the propagation of cracks, and the local crack tip stress intensity factor has been determined. Theoretically several authors have examined the process of dislocation emission from the crack tip by considering the elastic interaction between a crack and a dislocation. Transmission electron microscopy (TEM) was employed here to investigate the spatial configuration of dislocations emitted from crack tip in an Aluminum alloy (Al-1100) and Ni base superalloy (see Fig. 1). We showed that the mean position of dislocations of these zones are in reasonably good agreement with models of crack-dislocation configuration based on a continuum distribution dislocations ahead of the crack. However, these models fail to predict the actual number of dislocations emitted by the crack tip.

Existing theories assume a single source of dislocations at the crack tip, and fail to predict the total observed number of emitted dislocations. Using TEM, we showed, for the first time, that in fact several dislocation sources at the crack tip are operative, not one source. Our unique experimental approach established new insight as to the crack tip deformation and crack growth, and will enhance the predictability of crack growth models, which will be of obvious importance to Navy. We were able to verify using advanced TEM techniques that several dislocation sources are active at a crack tip, and that dislocations are emitted at several parallel (111) planes. We further observed that dislocations in the plastic zone are mostly screw type with no jogs, and are well separated and spread by dislocations cannot be explained by cross slip of dislocations. This conclusively shows that the microscopically "sharp" crack tip is in fact round at the atomic level, and the crack has a finite thickness.

Fatigue damage is one of the most insidious modes of mechanical failure of structures, but its underlying mechanism is still not fundamentally understood well enough to yield predictive models. Our efforts have provided significant new insight which, when incorporated in to fatigue predictive models, should provide more accurate crack growth predictions. The relationship between cyclic deformation behavior and changes in dislocation structures, lattice rotation and increase in residual stress is fundamental to the propagation of cracks. We have investigated the crack tip dislocations, lattice rotation and residual stress of aluminum alloys (see Fig. 2) under cyclic loading using TEM at different crack lengths corresponding to different stress intensity factors, a measure of loading criticality at the crack tip. Our TEM observations show the formation of sub-grains as well as high-density dislocation network structures with four fold and three fold nodes within the crack tip plastic zone (see Fig. 3). These networks form as a result of dislocation climb (movement of dislocation perpendicular to its plane), which requires supply of excess vacancies close to room temperature. Theories on fatigue damage have not considered such dislocation network formation. The incorporation of our discovery of this network structure into existing fatigue damage models should result in a step forward in predictive capability allowing for more reliable predictive capabilities and better estimates of component life.

Groundbreaking work includes the identification, for the first time, of significant lattice rotation in the plastic zone ahead of a fatigue crack at room temperature in Al 7075. Additionally, we observed significant grain growth in the plastic zone of fatigue cracks at room temperature in highly oriented Al 7075 alloys. Under Dr. Goswami's leadership, his research team has determined that this grain growth is associated with the formation of a very specific type of crystallographic texture component resulting from fatigue-assisted deformation. The previously accepted understanding was that grain growth is mostly thermally assisted. This finding is highly significant, because it shows that grain growth at room temperature can occur by lattice rotation and coalescence. In addition, we demonstrated that the residual stress increases gradually around the fatigue crack by $\approx 200\%$ for Al-7075 as we approach the crack (see Fig. 2). Such an increase in

residual stress cannot be explained by the change in dislocation density alone in both alloys due to crack growth. The results indicate that the deformation associated with the lattice rotation is a major factor controlling the stress. The lattice rotation that aligns 111 planes across grains, which is consistent with increase in shear-2 and near Cu texture (see Fig. 4), increases the stress. These findings provide new insights on the role of lattice rotation and dislocation configurations in the plastic region of a fatigue crack in determining the residual stress. This improves our understanding of fatigue damage and failure of metals, which is of vital importance to the Navy.

Fatigue crack growth tests were performed along short transverse (ST) direction in vacuum (< 6×10^{-6} Pa) background pressure at a cyclic load frequency of 10 Hz using a sine waveform and a load ratio of 0.10 on compact tension (CT) specimen, 80 mm in length, 60 mm in width and 10 mm in thickness, made of cold rolled-Al 7075-(T7) alloy. The T7 tempered condition corresponds to the heat treatments, such as solution annealing and overaging. Grains were observed to be elongated or pancake shaped parallel to the rolling direction. In order to study the characteristics of plastic zone and the dislocation configurations, the crack growth was arrested at a different crack lengths corresponding to a certain difference in stress intensity factor (ΔK) close to the Paris regime ($\Delta K = 13$) MPa m^{1/2}). X-ray diffractions were then obtained using a Rigaku 18kW generator and a high resolution powder diffractometer perpendicular to the crack length at number of locations in the CT specimen. The locations are close to the crack, and 5, 10, 15, 20 and 25 mm from the crack on both sides of the crack. The spot size of 1 mm wide window was used to collect the data at different locations. For EBSD, a JEOL JSM-7001F SEM operating at 30 kV and equipped with a TSL/EDX Hikari EBSD detector was used to acquire the data with a step size of 1.0 µm. Analysis of the EBSD data and grain size was performed with the TSL/EDX OIM Analysis-5.31 software package. To investigate the mechanical behavior, micro-indentation hardness tests were performed to obtain the hardness values as a function of distance from the crack with Vickers tip at 100g of load and dwell time of 15 seconds.

In addition, we came up with a prospective solution to the problem of heat sensitization in Al-Mg alloys, particularly in Al 5083 and Al 5456. These alloys are widely used for ship structural applications because of their lightweight, high strength and good weldability. However, structural parts manufactured with these alloys can fail catastrophically as a result of the formation of beta-Al₃Mg₂, known as Samson phase, at grain boundaries. Recently, we demonstrated that the Samson phase formation is suppressed in Al 5083 by alloying with small amounts of boron, which traps most of the Mg in solid solution as AlMgB₂ phase. This decreases the supersaturation level of Mg in Al, which is the driving force for the formation of grain boundary beta phase in Al 5083. We observe Cu-rich precipitates, instead of the beta phase, at grain boundaries upon extended annealing at 150°C. This was a significant finding as it provides a potential route to minimize the longstanding problem of ship structure sensitization. This is a major scientific achievement as it provides a potential route to minimize the longstanding problem of ship structure sensitization. This work has led to an NRL approved patent application.

Future Plan:

We plan to propose a program on manufacturing lightweight, high strength and corrosion resistant alloys for naval applications using recently developed materials processing and alloy additions at NRL. We came up with a prospective solution to the problem of heat sensitization in Al-Mg alloys, particularly in Al 5083 and Al 5456. These alloys are widely used for ship structural applications because of their lightweight, high strength and good weldability. However, structural parts manufactured with these alloys can fail catastrophically as a result of the formation of beta-Al₃Mg₂, known as Samson phase, at grain boundaries. Recently (FY17), we demonstrated that the Samson phase formation is suppressed in Al 5083 by alloying with small amounts of boron, which traps most of the Mg in solid solution as AlMgB₂ phase.



Fig. 1. (a) A bright-field TEM image close to the [110] zone showing dislocations ahead of a sharp crack and a dislocation free zone of Al 1100. The shape of the plastic zone is approximately elliptical. (b) TEM image showing the crack tip dislocations on Ni base super alloy, dislocation free zone and a plastic zone ahead of a crack.



Fig. 2. (a) A schematic showing the notch, crack and relative positions of experimental measurements on stress. (b) The EBSD image showing the grain orientation at either side of the crack. (c) Image showing the color coding of different orientations within a stereographic triangle containing 001, 110 and 111 poles. (d) The 113_{Al} peak at different ω . (e and f) Plots of d₁₁₃ as a function of sin² ψ at different locations from the crack for A-7075 and Al-1100, respectively. The slope of the best fit line is positive in most cases, suggesting that the stress is tensile in nature.



Fig. 3 (a) The residual stress as a function of distance from crack for Al-7075 and Al-1100. (b) The dislocation density as a function of distance from crack. (c) A bright-field TEM image showing the dislocation configuration close to the crack in Al-7075. (d) A bright-field TEM image showing the dislocation network configuration and relative dislocation free area close to the crack in Al-1100.



Fig. 4. (a) A series of x-ray diffraction (XRD) patterns obtained from Al 7075 at different locations, showing the relative variations of 111, 200, 220 and 113 Al peaks. We could see the intensity ratio of 111/200 peak increases by 175% as we move closer to crack, suggesting more 111 planes are aligned due to lattice rotation as compared to 200. (b) A series of XRD patterns obtained from Al-1100 at different locations, close to the crack, 5 mm and 25 mm from the crack. In this case, the intensity ratio of 113 to 220 drops significantly as we move closer to the crack, suggesting that the reduction of Cu texture, $\{112\}<111>$. (c) The ODF sections 25 mm away from crack parallel to φ 2 from 0-90° with an increment of 5° showing texture for Al-7075 (d) The ODF sections close to the crack for Al-7075. We observe considerable increase in texture components, such as shear-2 ($\{111\}<110>$) with ϕ 1, ϕ , ϕ 2 = 0, 55, 45, near Cu ($\{112\}<111>$) with ϕ 1, ϕ , ϕ 2 = 90, 35, 45.

Publications:

- 1. R. Goswami, C.R. Feng, S.B. Qadri and C.S. Pande, "Fatigue Assisted Grain Growth in Al Alloys", *Scientific Reports*, 7(1), 10179 (2017)
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- R. Goswami and C. S. Pande, (2018) "Crack-Dislocation Interactions Ahead of a Crack Tip". In: Hsueh CH. et al. (eds) Handbook of Mechanics of Materials. Springer, Singapore, DOI: <u>https://doi.org/10.1007/978-981-10-6855-3 52-1</u>
- R. Goswami and N. Bernstein, "Grain Boundary Precipitation and Fracture Behavior of Al-Cu-Li Alloys", in *Light Metals 2018*, STRN: NRL/CP/6350/17/468: DOI: https://doi.org/10.1007/978-3-319-72284-9_30
- 10. R. Goswami, C.R. Feng, S.B. Qadri and C.S. Pande, "A New Insight in the Deformation Process leading to Localized Changes in Texture, Dislocations and

Residual Stress Under Fatigue Loading", Materials Science and Engineering, communicated 2018, STRN: NRL/AO/6350/18/450

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Presentations:

- "Crack Tip Dislocations Under Static and Cyclic Loading, R. Goswami, S. B. Qadri,
 P. S. Pao and C. S. Pande, MS&T 2015, October 12-16, Pittsburg, PA (Invited)
- "Microstructure and Interfaces of Grain Boundary Al₂CuLi Plates of Al-3Cu-2Li" R. Goswami AND N. Bernstein, TMS 2015 Annual Meeting, Orlando, Fl, March 15-19, 2015 (Invited)
- "Crack Tip Dislocations Under Cyclic Loading, R. Goswami, S. B. Qadri, P. S. Pao and C. S. Pande, TMS-2015 Annual Meeting, Orlando, FL, March 15-19
- "TEM Studies of the Evolution of Dislocation Configurations Under Cyclic Loading in Al alloys", R. Goswami and C. S. Pande, TMS-2016 Annual Meeting, Nashville, TN, Feb 14-18, 2016 (Invited)
- "Effect of Ag and Mg Additions on the Nature Grain Boundary Precipitates and Fracture Behavior of Al-Cu-Li Alloys", R. Goswami and N. Bernstein, TMS-2016 Annual Meeting, Nashville, TN, Feb 14-18, 2016 (Invited)
- "Effect of Ag and Mg on the Microstructure and Interfaces of Grain Boundary T₁ Plates of Al-Cu-Li Alloy", R. Goswami, MS&T, 2015, Oct 05-08, Columbus, OH
- 7. "Effect of Grain Boundary Microstructural Features on the Fracture Behavior of Al-

Li alloys", R. Goswami, International Conference on Environmental Damage on Structural Materials, Cork, Ireland, May 29-June 03, 2016

- "Fatigue Mediated Lattice Rotation of Plastic Zone in Al Alloys", R. Goswami S.B.Qadri and C. S. Pande, TMS-2017 Annual Meeting, San Diego, CA, Feb 26-March 02, 2017
- "Microstructures and Properties of Al/Al₂O₃ Multilayers" R. Goswami and C. S. Pande, MS&T, 2016, Oct 23-27, Salt Lake City, UT
- "Grain Boundary Precipitation and Fracture Behavior in Al-Cu-Li Alloys", R. Goswami and N. Bernstein, *TMS 2018 Annual Meeting*, Phoenix, AZ, March 11-15, 2018: STRN: NRL/OP/6350/18/194
- "Fatigue-Assisted Discontinuous Grain Growth in Al alloys", R. Goswami, C.R. Feng, S. B. Qadri and C.S. Pande, *TMS 2018 Annual Meeting*, Phoenix, AZ, March 11-15, 2018: STRN: NRL/OP/6350/18/195
- "Suppression of Samson Phase Formation in Al-Mg Alloys by Boron Addition", R. Goswami and S.B. Qadri, *MS&T 2017*, Pittsburgh, PA, October 11, 2017, STRN: NRL/AO/6350/17/238
- R. Goswami and N. Bernstein, "Grain Boundary Precipitation and Fracture Behavior of Al-Cu-Li Alloys" NRL External Review, June 22 2018: STRN: NRL/OE/6350/18/433
- 14. R. Goswami, "Fatigue assisted lattice rotation in Al Alloys", NRL External Review, June 22 2018: STRN: NRL/OE/6350/18/432
- 15. R. Goswami, "Fatigue assisted lattice rotation and grain growth in Al alloys", International Conference on Fatigue Damage on Structural Materials, Cape Cod-Hyannis, MA, September 2018: STRN: NRL/AP/6350/18/310

Patent:

 R. Goswami and S. B. Qadri, "Suppression of Samson Phase Formation in Al-Mg Alloys by Boron Addition", Navy Case # 105,832-US1 (US Patent application submitted January 2018): STRN: NRL/OE/6350/18/396