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14. ABSTRACT Residual stress plays an important role in mechanical properties of polycrystalline materials, including deformation, fracture, corrosion and fatigue, etc. The effects become more significant when the grain size reaches nanometer scales where the local stress level could reach gigapascal. Residual stress in polycrystalline materials with grain size of microns or larger can be acquired via x-ray or neutron scattering, in conjunction with finite element continuum modeling. Below a few hundred nanometers, however, these methods fail. How to obtain residual stresses at these small scales remains an open issue.					
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Report Title

Final Report for ARO sponsored research (ARO-W911NF-12-1-05869)

ABSTRACT

Residual stress plays an important role in mechanical properties of polycrystalline materials, including deformation, fracture, corrosion and fatigue, etc. The effects become more significant when the grain size reaches nanometer scales where the local stress level could reach gigapascal. Residual stress in polycrystalline materials with grain size of microns or larger can be acquired via x-ray or neutron scattering, in conjunction with finite element continuum modeling. Below a few hundred nanometers, however, these methods fail. How to obtain residual stresses at these small scales remains an open issue.

A more challenge issue is how to obtain residual stresses associate with different microstructural components, such as each grain boundary, triple junction line, or a grain, which have even smaller length scales, usually at angstrom or less than a nanometer. This question is critical to understanding the effects of the residual stress on material properties in a quantitative fashion. As far as we know, there is no prior work on this topic. As a result, few results are available for the local information in polycrystalline materials.

The purpose of this work is to connect the mechanical properties of nanocrystalline Mg and Mg alloys to the residual stresses and furthermore, determine quantitatively the residual stress in nanocrystalline Mg, Mg alloys and other hcp metals. This work is of particular importance since hcp metals and alloys are known to have strong structural and mechanical anisotropy. They tend to form textures which make their mechanical properties strongly susceptible to internal stress and vice versa.

Our goal in this work is therefore to quantitatively determine the internal stress and link the internal stress to microstructural components such as grain boundaries, triple junctions, and grains to residual stresses. Therefore, quantitative relations between the microstructures and mechanical properties can be established via residual stress.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received

Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	<u>Discipline</u>
Shangduan Wu	1.00	
FTE Equivalent:	1.00	
Total Number:	1	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	
Tao Xu	0.50	
FTE Equivalent:	0.50	
Total Number:	1	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	<u>National Academy Member</u>
Mo Li	0.10	
FTE Equivalent:	0.10	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	
FTE Equivalent:		
Total Number:		

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

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Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

The key challenge in this work is to develop effective numerical techniques to characterize microstructures for hcp metals that have high structural and elastic anisotropy and far less number of slip systems. Another one is how to quantitatively measure the internal stresses and connect them to the microstructural components.

We developed a bottom-up approach to compute residual stresses for nc materials through atomistic stress calculation. In conjunction with the digital microstructure classification method we developed early, we are able to obtain various residual stresses from atomic scale, and for all microstructural components.

Technology Transfer

Final Report for ARO sponsored research (ARO-W911NF-12-1-05869)

Mo Li

School of Materials Science and Engineering
Georgia Institute of Technology

I. (a) Project Report Period:

August 2012 – July 2013 (12 months)

I. (b) Project Participants:

Mo Li – PI

Tao Xu – part-time postdoc

Shangduan Wu – Graduate student in Materials Science

II. Journal Publications:

1. Y. B. Guo, M. Li, "Hierarchical Dislocation Nucleation Controlled by Internal Stress in Nanocrystals" *Applied Physics Letters* **102**, 241910, 2013.
2. Y. B. Guo, T. Xu, and M. Li, "Generalized Type III Internal Stress from Microstructures in Nanocrystalline Materials." *Acta Materialia* **61**, 4974, 2013.

* Papers in preparation and to be submitted:

3. T. Xu and M. Li, " Microstructure and Diffraction Simulation of Nanocrystalline Materials" *In preparation*.
4. S. D. Wu and M. Li, "Internal Stress in Nanocrystalline Magnesium." *In preparation*.
5. T. Xu and M. Li, "Deformation and Fracture Mechanism of Nanocrystalline Magnesium: The Effects of Misorientation." *In preparation*.

III. Research accomplishments and description:

A. Purpose and goal:

Residual stress plays an important role in mechanical properties of polycrystalline materials, including deformation, fracture, corrosion and fatigue, etc. The effects become more significant when the grain size reaches nanometer scales where the local stress level could reach gigapascal. Residual stress in polycrystalline materials with grain size of microns or larger can be acquired via x-ray or neutron scattering, in conjunction with finite element continuum modeling. Below a few hundred nanometers, however, these methods fail. How to obtain residual stresses at these small scales remains an open issue.

A more challenge issue is how to obtain residual stresses associate with different microstructural components, such as each grain boundary, triple junction line, or a grain, which have even smaller length scales, usually at angstrom or less than a nanometer. This question is critical to understanding the effects of the residual stress on material properties in a quantitative fashion. As far as we know, there is no prior work on this topic. As a result, few results are available for the local information in polycrystalline materials.

The purpose of this work is to connect the mechanical properties of nanocrystalline Mg and Mg alloys to the residual stresses and furthermore, determine quantitatively the residual stress in nanocrystalline Mg, Mg alloys and other hcp metals. This work is of particular importance since hcp metals and alloys are known to have strong structural and mechanical anisotropy. They tend to form textures which make their mechanical properties strongly susceptible to internal stress and vice versa.

Our goal in this work is therefore to quantitatively determine the internal stress and link the internal stress to microstructural components such as grain boundaries, triple junctions, and grains to residual stresses. Therefore, quantitative relations between the microstructures and mechanical properties can be established via residual stress.

B. Key Challenges:

The key challenge in this work is to develop effective numerical techniques to characterize microstructures for hcp metals that have high structural and elastic anisotropy and far less number of slip systems. Another one is how to quantitatively measure the internal stresses and connect them to the microstructural components.

C. Innovation and Unique Contributions:

We developed a bottom-up approach to compute residual stresses for nc materials through atomistic stress calculation. In conjunction with the digital microstructure classification method we developed early, we are able to obtain various residual stresses from atomic scale, and for all microstructural components.

D. Internal stress in nanocrystalline Mg:

(a) Calculation methods:

hcp Mg nanocrystalline samples made with different grain sizes were prepared. The mean grain size ranges from 9 nm to 26 nm that contains about

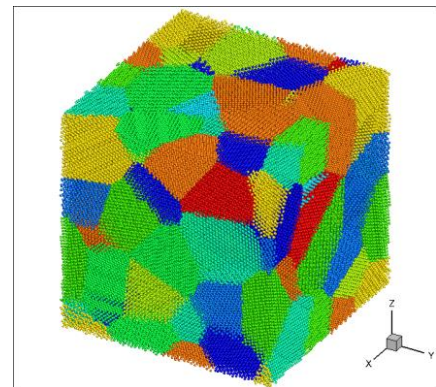


Figure 1 A nc-Mg sample containing 50 grains and with 8.9 nm mean grain diameter. The different color represents different crystal orientation.

30-50 million atoms. An nc-Mg sample is shown in Figure 1. The polycrystalline sample is then dissected into different microstructure components or attributes of grain cell (GC), grain boundary (GB), triple junction (TJ) and vertex point (VP) as shown in Figure 2.

The internal stress is calculated from the “atomic stress” which is obtained from the interatomic potential via the following relation,

$$\sigma_{\alpha\beta}(i) = \frac{1}{\omega_i} \left[-m_i v_\alpha^i v_\beta^i + \sum_{i < j}^N \left(-\frac{1}{r_{ij}} \frac{\partial U}{\partial r_{ij}} \right) r_\alpha^{ij} r_\beta^{ij} \right],$$

the atomic distance between two atoms, $\vec{r}_{ij} = \vec{r}_i - \vec{r}_j$,

its velocity, \vec{v}_i , and the interatomic interaction U . α and β are Cartesian components. To represent atomic stress in a simpler manner, we use the two eigenvalues of the stress, one is the pressure or the

trace $\sigma_{hy}(i) = \frac{1}{3} [\sigma_{xx}(i) + \sigma_{yy}(i) + \sigma_{zz}(i)]$ and another is the von Mises shear stress,

$$\sigma_{vm}(i) = \left\{ \frac{1}{2} [(\sigma_{xx}(i) - \sigma_{yy}(i))^2 + (\sigma_{xx}(i) - \sigma_{zz}(i))^2 + (\sigma_{zz}(i) - \sigma_{yy}(i))^2 + 6(\sigma_{xy}^2(i) + \sigma_{zy}^2(i) + \sigma_{zx}^2(i))] \right\}^{1/2}$$

The residual stress is further classified as type I, II and III stress according to their self-equilibrating length scales. *Type I* stress is that averaged over the entire sample; *type II* is associated with atoms inside the GCs only; *type III* is for those associated with the GC, GB, TJ and VP. In general the different types of residual stress can be

written as $\sigma_{\alpha\beta}^{type}(m) = \frac{1}{\Omega_m} \sum_{i=1}^{N_m} \omega_i \sigma_{\alpha\beta}(i)$, where Ω_m and N_m are the volume and the

number of atoms associated with the self-equilibrating length or object. For example, if Ω_m is the sample volume and N_m is the total number of atoms in the system, the stress is type I stress. If Ω_m is the volume of a microstructure component such as a grain boundary or a triple junction which are labeled as m , and ω_i is the atomic volume of atom i , and N_m is the number of atoms in the grain boundary or a triple junction, then the stress is type III stress.

(b) Grain size and misorientation effect:

Because the reduction of the grain size introduces more disorder at the grain boundaries, the grain size is expected to play an important role in the residual stresses. On the other hand, misorientation in the strong mechanically anisotropic material such

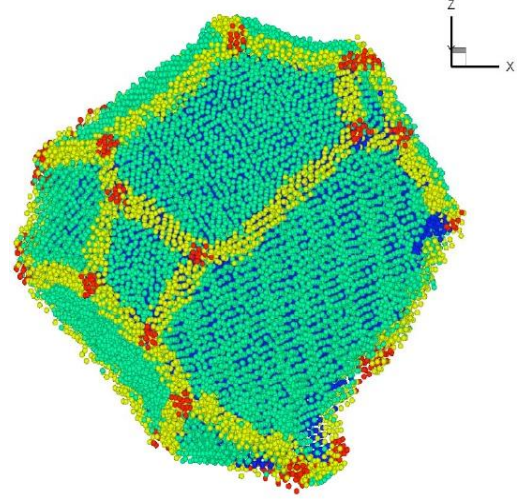


Figure 2 A grain taken from the nc-Mg sample shown in Figure 1. The GCs, GBs, TJs and VPs associated with this grain are marked by blue, green, yellow and red atoms respectively.

as Mg would affect the movement of atoms in sample preparation and deformation; we therefore expect to see certain effect from it on residual stresses. Indeed, our work shows that both the grain size and grain boundary misorientation play important roles in residual stresses.

Figure 3 shows the distribution of the type II von Mises shear residual stress of nc-Mg samples with various grain size and two different misorientations at 10 and 40 degrees. We observe that (1) The smaller the grain size, the larger the residual stress; (2) the larger the variance (spread) of the residual stress; (3) the smaller the misorientational angle, the larger the residual stress and larger its variance. Besides, the distribution of the residual stress follows a lognormal distribution. Figure 4 summarize the results.

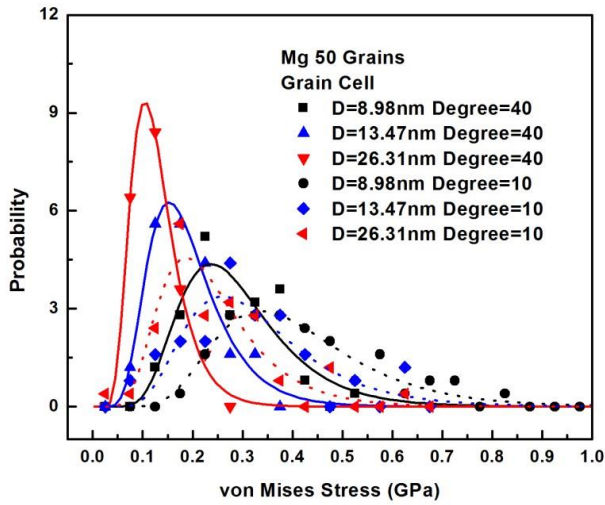


Figure 3 The distribution of the type II von Mises residual stress in nc-Mg samples. Two variables are used here: the mean grain size and the misorientation angle.

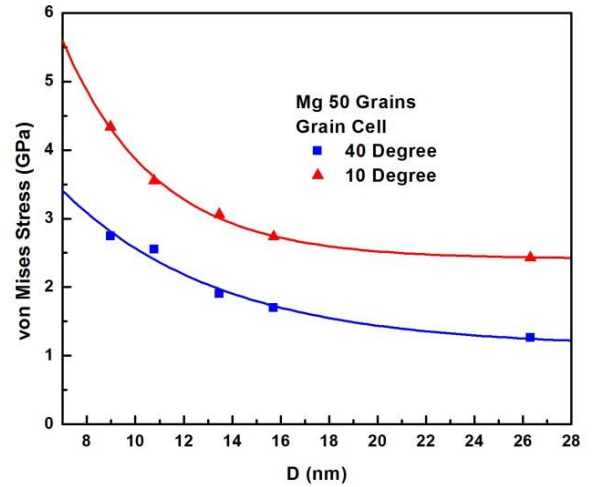


Figure 4 The mean values of the type II von Mises residual stress in nc-Mg samples vs. grain size and the misorientational angle.

(c) Type III residual stresses

Type III as well as IV residual stresses associated with different microstructural components are calculated for nc-Mg. As shown in Figure 5, the type III von Mises residual stress associated with the grain boundary is smallest, next comes the triple junction and vertex point. Figure 6 shows the type III residual pressure. As a comparison, we also plotted the type IV, or atomic stress, in the sample and the type II residual stress in the grain cell. For all type III stresses, their variances or the spreads also follow the same trend: the GB is the smallest, which follows by the TJ and VP.

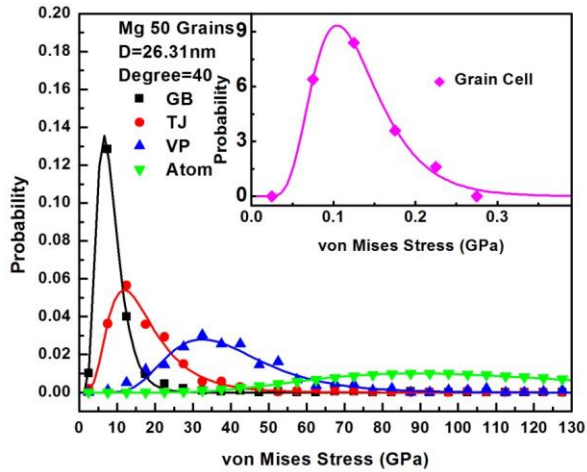


Figure 5 The distribution of the type III von Mises residual stress in nc-Mg samples. Two variables are used here: the mean grain size =26.31 nm and the misorientation angle = 40 degree. Type IV stress or atomic stress is also plotted.

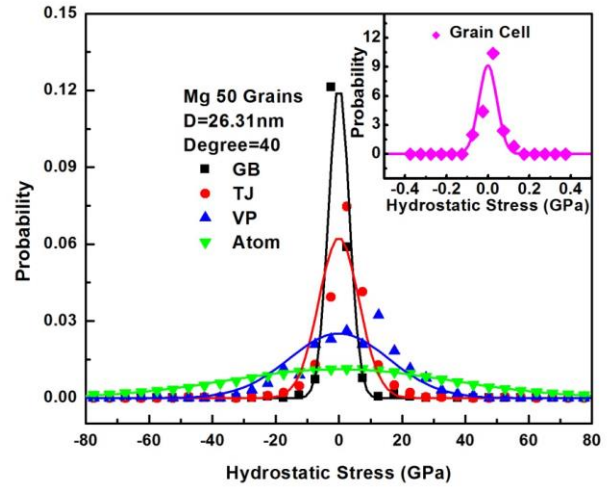


Figure 6 The distribution of the type III residual pressure in nc-Mg samples. Two variables are used here: the mean grain size =26.31 nm and the misorientation angle = 40 degree. Type IV stress or atomic stress is also plotted.

Figure 7 ad 8 show the dependence of the type III residual stress on grain size and misorientation angles. As seen for type II residual stress, the larger the grain size, the smaller the type III residual stress and narrower the variance; conversely, the smaller the misorientational angle, the larger the type III stress and larger the variance.

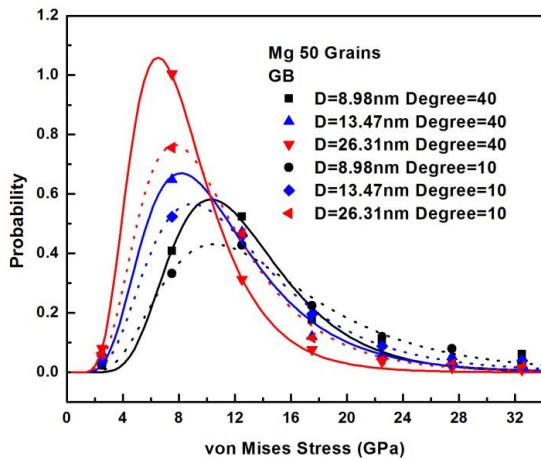


Figure 7 The distribution of the type III residual von Mises stress for grain boundary in nc-Mg samples. Two variables are used here: the mean grain size and the misorientation angle.

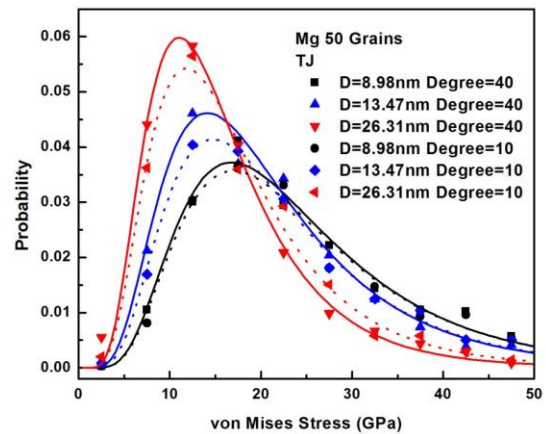


Figure 8 The distribution of the type III residual von Mises stress for triple junction in nc-Mg samples. Two variables are used here: the mean grain size and the misorientation angle.

Figure 9 summarizes the results for GB, TJ and VP. The results show that the type III von Mises residual stress for GB, TJ and VP all increase with the decreasing grain size and increasing misorientational angle. But the type III pressure exhibits the opposite trend: the smaller the grain size, the smaller the pressure (Figure 10).

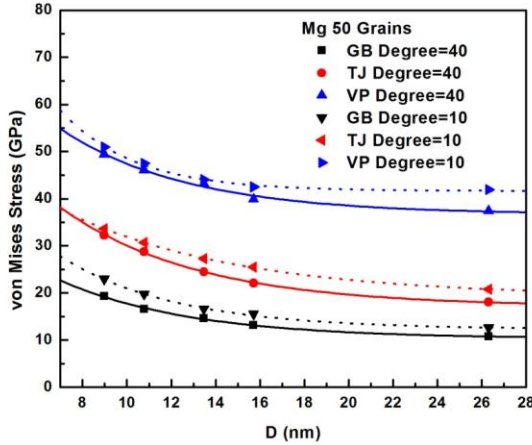


Figure 9 The dependence of the mean type III von Mises mean residual stress on grain size and misorientational angle for nc-Mg samples.

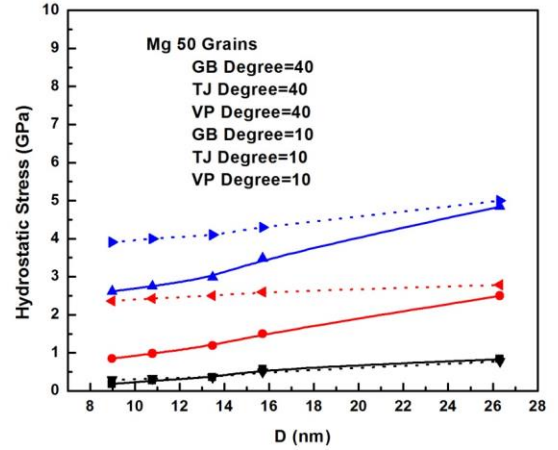


Figure 10 The dependence of the mean type III mean residual pressure on grain size and misorientational angle for nc-Mg samples.

IV. Summary and discussion

In the past year, we have made significant progress in acquiring the residual stresses of all types in nc-Mg. We have performed some of the most extensive atomistic calculations with 30-50 million atoms for Mg as its interatomic interactions are long-range and also it has strong structure anisotropy. The information obtained is the first of this kind for nc-Mg.

In particular, we observed that grain size as well as GB misorientation affects residual stress tremendously. The grain size reduction leads to increase in all types of residual stresses. But the misorientation shows a counter-intuitive result, that is, the larger the misorientation angle, the smaller the residual stress. This trend is opposite to that observed in more structurally isotropic metals such as Cu and Ni. We may explain this trend by the strong mechanical anisotropy in hcp metals such as Mg. Since the number of the degrees of freedom for atoms to move or relax in an hcp metal is limited by the strong mechanical/structural anisotropy, when the misorientational angles are reduced from nearly random ($\sim 46^\circ$) to smaller angles (say, 10°), the available degrees of freedom for the atoms to move become smaller. In other words, if two nearby grains have the nearly the same orientation, the possibility of the atoms in the two grain to move in order to accommodate the grain boundary disorder becomes smaller, which is not the case in the less isotropic fcc metals where atoms have more opportunities to relax in all possible

directions. Therefore, similar as the easy texture formation in hcp nanocrystals, the strong misorientation dependence of the residual stress is one of its features that we discovered in this work.

V. Future work

A. Connecting the residual stress to deformation mechanisms

With the information and ability to quantitatively measure and characterize the residual stress, the next question we ask is what the connection between the residual stresses to the general deformation mechanisms in nc-Mg. Our preliminary work (see publication 1 and 2 in section II) shows that residual stress has a strong influence on dislocation nucleation and propagation. In structurally more isotropic fcc crystals, the presence of large type III residual stress at the VP and TJ promotes easy nucleation of dislocations while GB and GC remains relatively dormant until large deformation occurs. The hierarchical dislocation formation provides us with a rare opportunity to modify the microstructure and processing nc materials in order to overcome the brittleness.

For nc-Mg, the lack of intrinsic slip systems motives us to look for different rules played by the residual stresses than that in fcc metals. One possibility is how the residual stress promote/demote the twins; the second possibility is a more profound correlated motion of grains as compared with that of fcc metals where the easy slip does not require grain to move together in a correlated fashion.

Figure 11 shows some of our preliminary results in modeling the deformation and fracture of an nc-Mg with different misorientational angles. The sample is under tension. As seen, the fracture in both cases occurs almost predominantly on the grain boundaries. It seems that misorientation has little effect in changing the fracture mode. The brittle nature of the nc-Mg is not become alleviated by simply reduction of the grain size. Obviously more microstructure engineering is needed.

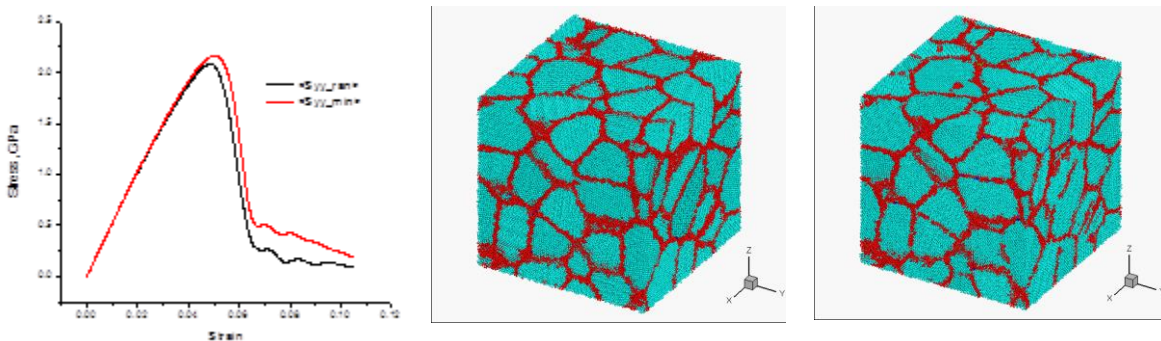


Figure 11 *Left:* The stress-strain curve for an nc-Mg sample with a 40° (red) and 10° (black) misorientation angle. *Center:* The atomic structure at yielding of the sample with 40° misorientational angle under tension. *Right:* The atomic structure at yielding of the sample with 10° misorientational angle under tension.

B. Constitutive models

All the efforts made or to be made have one final goal, that is, for us to establish reliable and physically justified constitutive relations for nc-Mg and other hcp nanocrystalline materials. Although this is a long-term goal, nevertheless we should pay close attention to this perspective in our work on residual stress and interface stress. The role of the residual stress, and in general, the interface stress, is to set the backdrop of the stress state for nc-hcp metals where defect formation, deformation process and fracture will take place. We plan to embark on this topic in the next phase with the possible support from ARO.