REPORT DOCUMENTATION PAGE

AFRL-SR-BL-TR-01-

0508

Public reporting burden for this collection of information is estimated to average 1 hour per response, inclu
gathering and maintaining the data needed, and completing and reviewing the collection of information. Se
collection of information, including suggestions for reducing this burden, to Washington Headquarters Servi
Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Pap

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REP
	01-OCT-01	Final Report 04-APR-01 TO 31 JUL 01

4. TITLE AND SUBTITLE
EXPERIMENTAL DETERMINATIN OF DISLOCATION CORE STRUCTURES BY HRTEM

5. FUNDING NUMBERS
F49620-98-1-0208

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AFOSR/NA
801 NORTH RANDOLPH STREET
ROOM 732
ARLINGTON, VA 22203-1977

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

20011012 006

12a. DISTRIBUTION AVAILABILITY STATEMENT

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13. ABSTRACT *(Maximum 200 words)*

The overarching scientific goal of this study is to use start-of-the-art TEM techniques to experimentally characterize dislocation core structures in metals and alloys at the atomic scale. The past year has involved the culmination of efforts on intermetallic alloys and pure fcc metals and the development of new techniques for the study of bcc metals and alloys. Our work on (Ni, Fe) Ge indicated that changes in mechanical behavior associated with Fe additions are related to variations in dislocation core geometry and its effect on dislocation mobility. These findings have both motivated and been found to be in agreement with first principles based calculations of dislocation core structures in these alloys. In a related study, our weak-beam TEM and HREM experimental observations of dislocation core structures in fcc gold and iridum have been shown to be in excellent agreement with theoretical predictions for these metals. More recent efforts have focused on developing the techniques for characterizing dislocation core structures in bcc metals. Direct HREM observations of dislocation cores and the derivation of localized electron structure data from electron energy loss spectra (EELS) measurements are long term goals, and preliminary steps toward these challenging tasks are underway.

14. SUBJECT TERMS
EXPERIMENTAL DETERMINATIN OF DISLOCATION CORE STRUCTURES BY HRTEM

15. NUMBER OF PAGES
7

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

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UNIVERSITY

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August 17, 2001

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EXPERIMENTAL DETERMINATION OF DISLOCATION CORE STRUCTURES BY HRTEM

AFOSR Grant No: F49620-98-1-0208

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Abstract

The overarching scientific goal of this study is to use state-of-the-art TEM techniques to experimentally characterize dislocation core structures in metals and alloys at the atomic scale. The past year has involved the culmination of efforts on intermetallic alloys and pure fcc metals and the development of new techniques for the study of bcc metals and alloys. Our work on $(Ni,Fe)_3Ge$ indicated that changes in mechanical behavior associated with Fe additions are related to variations in dislocation core geometry and its effect on dislocation mobility. These findings have both motivated and been found to be in agreement with first-principles based calculations of dislocation core structures in these alloys. In a related study, our weak-beam TEM and HREM experimental observations of dislocation core structures in fcc gold and iridium have been shown to be in excellent agreement with theoretical predictions for these metals. More recent efforts have focused on developing the techniques for characterizing dislocation core structures in bcc metals. Direct HREM observations of dislocation cores and the derivation of localized electron structure data from electron energy loss spectra (EELS) measurements are long term goals, and preliminary steps toward these challenging tasks are underway.

Research Objectives

The development and use of theoretical and computationally intensive tools have a very important role to play in the future of materials science and engineering. Judicious use of these techniques as design tools provide very powerful opportunities for understanding and modeling atomic level processes, and the development of truly multi-scale modeling techniques holds great promise for enhancing our understanding of the processing-structure-property-performance relations that govern alloy design and materials behavior in general. One example of efforts to promote more efficient alloy design is the work that is currently being pursued to both theoretically predict and experimentally measure the effect that dislocation core geometry has on the macroscopic strength of metals and alloys. Recent advances in theoretical modeling appear very promising but are currently inhibited by the limited amount of experimental data that is available to verify and test their predictions. The primary objective of this study is to develop the techniques for and provide quantitative observations of dislocation cores in pure metals and alloys. These detailed measurements are intended to provide benchmarks for theoretical studies (most directly through collaborations with Drs. Woodward and Rao in the Materials Directorate, Wright Laboratory, Wright-Patterson Air Force Base and Professor Freeman at Northwestern University) and to establish a link between theoretical, computational and experimental approaches to determining the effect of the core structure on the mobility of dislocations in metals and alloys.

Experimental Results and Discussion

Intermetallic alloys. Ni_3Ge exhibits a yield strength anomaly, while the yield strength of Fe_3Ge exhibits a normal decline with temperature. There is a gradual transition from anomalous to normal behavior as Fe content increases, but a dramatic strengthening for 77 K deformation has also been noted to occur in these alloys as a result of increasing Fe content. The combined use of TEM and image simulations has facilitated identification of the operative deformation mechanisms and allowed for a quantitative measure of superdislocation dissociations. A transition from octahedral glide and Kear-Wilsdorf locking to cube glide of superdislocations has been observed to coincide with increases in either deformation temperature or Fe content. The low temperature strengthening has been correlated with enhanced cross-slip, which is aided by a significant lowering of the cube plane antiphase boundary energy with increasing Fe content. It was further proposed that the strengthening and the transition to cube glide are promoted by an increase in the complex stacking fault energy, which enhances both cross-slip and cube plane mobility. This energy was measured for Ni_3Ge but could not be obtained for Fe_3Ge and thus no experimental comparison is available. First-principles calculations have, however, been used to show that the screw dislocation core structure in Fe containing alloys should result in increased cross-slip locking and mechanical strengthening [1]. This theoretical finding is fully consistent with the experimental results and provides a more complete understanding of the relationship between dislocation core geometery and mechanical behavior in this important class of alloys. The experimental portion of this work has recently been published in [2].

Fcc metals. A combined weak-beam TEM and HREM study has resulted in the quantification of extremely small dislocation dissociation distances, as well as the calculation of stacking fault energies for pure single crystalline gold and iridium. It has been found that screw and 60° dislocations in gold are dissociated by 0.8 and 3.25 nm, respectively, while in iridium, the corresponding distances are 0.8 and 1.25 nm. Hence, the first-principles prediction that gold and iridium display similar dislocation dissociation behavior appears to be true, as the dissociation widths for the screw orientation is the same in both metals. These HREM results agree with weak-beam measurements of dissociation width, and all values fall on a common curve relating dislocation character to splitting distance, as calculated using anisotropic elasticity. The value reported here for gold, 33 mJ/m^2, agrees well with previous findings (Jenkins 1972), while the energy for iridium, 420 mJ/m^2, is a new value that has not previously been determined. These results offer ideal benchmarks for first-principles calculations, which can in turn be used to predict material parameters such as the unstable stacking energy, and thus illuminate the brittle versus ductile behavior of iridium and gold. This work has recently been published [3,4] and has been found to be in very good agreement with the first-principles based predictions of Freeman et al. [5].

Bcc metals and alloys. Duesbery and Vitek [6] have related the behavior of bcc metals to non-linear core effects and the intrinsic-lattice coupling that occurs as a screw dislocation moves in the bcc crystal structure. They have proposed that Schmid's Law violations and Peierls stress variations are the result of: (1) the intrinsic geometry of the dislocation core and its coupling to the generalized stacking fault (γ-surface) and (2) small edge components in the screw dislocation core structure that are strongly influenced by the presence of non-glide components of the applied stress. Their atomistic calculations, based on Finnis-Sinclair interatomic potentials, indicated that Group VB and VIB metals show distinctly different dislocation core properties. More recent first-principles calculations, see for example Rao and Woodward [7], predict that isolated screw dislocations are evenly spread onto three conjugate (110) planes – with 6-fold symmetry- for both Mo and Ta, and do not support the six-fold / three-fold classification that was put forth as a result of the atomistic calculations. The current focus of our study is aimed at obtaining reliable experimental observations that can be used as benchmarks for both the first-principles and atomistic simulations.

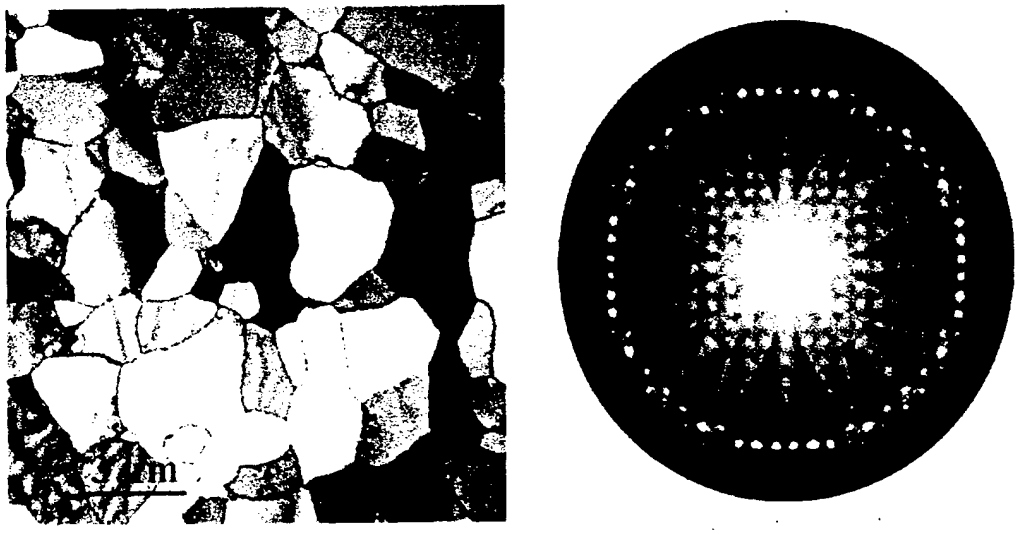

Fig. 1: Bright field TEM image and a [100] zone diffraction pattern of polycrystalline Mo.

Polycrystalline Mo with a purity of 99.5% was annealed at 1000°C for 10 minutes and cut to 0.3 mm thick slices by electro-discharge machining. TEM samples were electropolished in a solution of 10% sulfuric acid, 30% butoxyethanol and 60% methanol (volume percentage) at -30°C and plasma-cleaned before each TEM session. A Phillips CM-300 FEG TEM equipped with Gatan electron energy loss spectroscopy (EELS) detector, operated at 300 kV, was employed to investigate the microstructure and EELS spectrum. The microstructure of the poly-Mo is shown in Fig. 1. The alloy has very fine grains with size ranging from 1 to 4 um. Selected area electron diffraction patterns indicated that most of the grain boundaries are small angle grain boundaries with mis-orientation below 2 degrees.

A typical low energy loss spectrum of Mo is exhibited in Fig. 2, consisting of zero loss (ZL) peak, plasmon and $N_{4,5}$ edge. From ZL peak, it is calculated that the energy resolution is 1 eV in the present study. The effect of multiple scattering in the spectrum is small and the main contribution to the feature on the high energy tail of the plasmon is the $N_{4,5}$ transition of $3d$

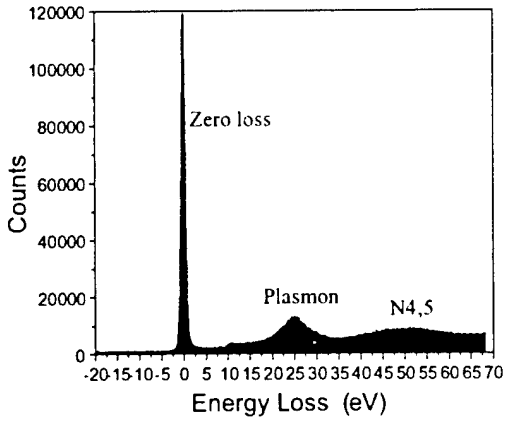

Fig. 2: A typical low energy loss spectrum of Mo with a energy resolution of 1 eV.

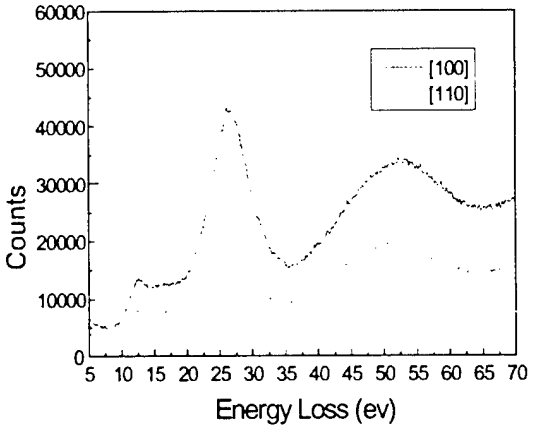

Fig. 3: Low energy loss spectra of Mo with different crystal orientations.

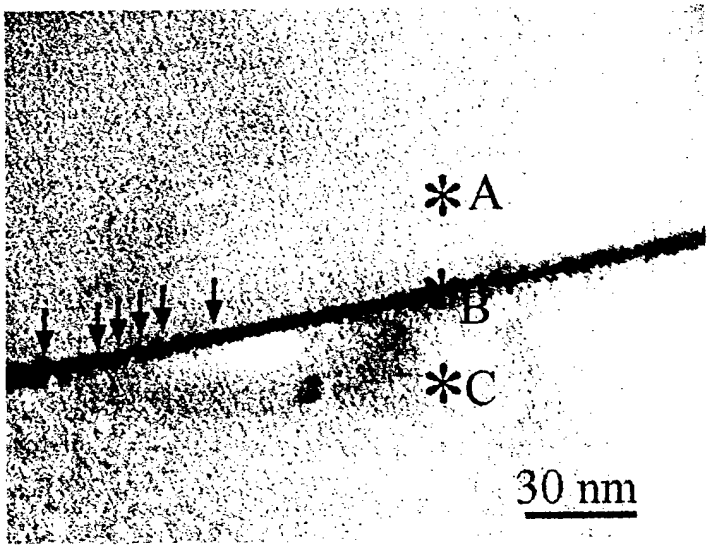

Fig. 4: A low angle grain boundary. Arrowheads indicate the GB dislocations. Stars(*) show the locations for EELS probe.

electrons. As an initial step to exploring the electronic structure of crystal defects in bcc metals by TEM EELS, we first investigated the effect of low angle grain boundaries with dislocation structure on the low energy loss. To probe the grain boundary, the diameter of electron beam is focused to 1 nm. We found that the low energy loss of Mo is sensitive to the crystal orientation, see for example the comparison of spectra for grains orientated along [001] and [111] in Fig. 3, and as a result three points are detected during each series, at grain boundaries and within the grains on either side of the boundary, see Fig. 4. By comparing the two spectra from the two grains, the possible influence of crystal orientation changes across the boundaries on energy-loss features can be ruled out. The preliminary results are shown in Figs. 5 and 6. It is encouraging to note that the spectra from the perfect crystals of the two-side grains generally match each other well, and that the boundary spectra show obvious differences in both peak intensity and width.

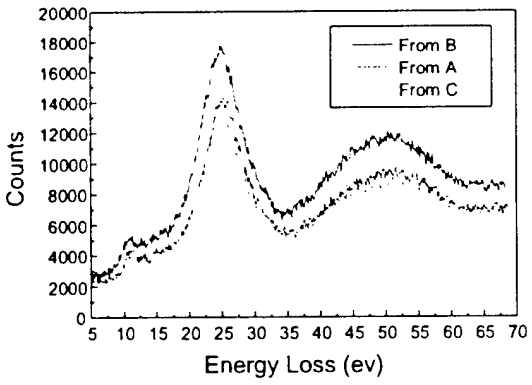

Fig. 5: Low energy loss spectra cross a grain boundary of Mo with the crystal orientation near [100].

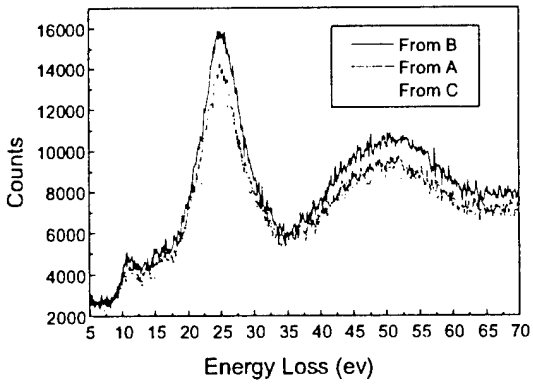

Fig. 6: Low energy loss spectra cross a grain boundary of Mo with the crystal orientation near [120].

It is believed that the major contribution to the low energy loss spectra comes from the excitation of electrons in the valence bands to empty states in the conduction bands [8,9]. There are few studies on plasmon behavior of the 4d metals [10,11], and such studies have demonstrated core polarization near the end of the series. Core polarization induces a decreasing shift in the plasmon frequency, and results from the movement and narrowing of the 4*d* band relative to the free electron conduction band. The present results clearly show that electronic structure of grain boundaries is different from that of the perfect crystal. However, 4*d*-5*sp* inter-band transitions generally complicate the low loss structure of the 4*d* series and make the quantitative interpretation of the plasmon behavior more difficult. Here, as an ambitious explanation, the grain boundary effect observed in this work may be due the free electron density change. In the future work, quantitative theoretical analysis will be performed to deeply understand the electronic structure of the dislocation grain boundaries.

Acknowledgment/Disclaimer

This work is sponsored (in part) by the Air Force Office of Scientific Research, USAF, under grant number F49620-98-1-0208. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Air Force Office of Scientific Research or the U.S. Government.

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Personnel Supported

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Publications

T.J. Balk and K.J. Hemker, "Experimental Observations of Dislocation Core Structures in Gold and Iridium", *Material Science and Engineering A*, **309-310** (2001) 108-112.

T.J. Balk and K.J. Hemker, "HREM Observations of Dislocation Cores in Single Crystalline Gold and Iridium", *Phil. Mag. A*, **81** (2001) 1507-1531.

T.J. Balk, Mukul Kumar and K.J. Hemker, "Influence of Fe Substitutions on the Deformation Behavior and Fault Energies of Ni_3Ge-Fe_3Ge $L1_2$ Intermetallic Alloys", *Acta materialia*, **49** (2001) 1725-1736.

Awards Received

Kevin Hemker awarded the 2001 ASM Silver Medal
Kevin Hemker promoted to full professor at Johns Hopkins University July 1, 2001.

Transitions

None this year