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RESEARCH PROJECT INITIATION

Date: May 24, 1973

Project Title: Effects of Interface Phenomena on Mechanical Behavior of Metals

Project No: E-19-616*

Co-Principal Investigator Dr. E. A. Starke (with Dr. B. R. Livesay, EES)

Sponsor: Air Force Office of Scientific Research (AFSC)

Agreement Period: From April 1, 1973 Until December 31, 1973

Type Agreement: Grant No. AFOSR-71-2064 (Mod. C)

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See A-1319 (EES)

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*Sub-project under A-1319 (EES), Dr. B. R. Livesay - Co-Principal Investigator, Budgeted for $14,961; total est. cost (AFOSR) is $30,000.

Assigned to: School of Chemical Engineering

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RESEARCH PROJECT TERMINATION

Date: 2/28/74

Project Title: "Effects of Interface Phenomena on Mechanical Behavior of Metals"

Project No: E-19-616

Principal Investigator: Dr. E. A. Starke

Sponsor: Air Force Office of Scientific Research

Effective Termination Date: December 31, 1973

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Engineering Experiment Station

PROJECT INITIATION

Date: 3/23/71

Project Title: Effects of Interface Phenomena on Mechanical Behavior of Metals

Project No.: A-1319

Project Director: Dr. E. A. Starke and Mr. B. R. Livesay

Sponsor: Air Force Office of Scientific Research

Effective . . . . . . April 1, 1971 . . . . Estimated to run until: . . . March 31, 1972 . . . .

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GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station

PROJECT TERMINATION

Date March 6, 1974

PROJECT TITLE: "Effects of Interface Phenomena on Mechanical Behavior of Metals"

PROJECT NO: A-1319

PROJECT DIRECTOR: Dr. B. R. Livesay

SPONSOR: Air Force Office of Scientific Research

TERMINATION EFFECTIVE: February 28, 1974

CHARGES SHOULD CLEAR ACCOUNTING BY: March 31, 1974

REMAINING TERMINATION ACTION: Final Fiscal Report (due no later than 3/31/74)
Patent Report

Project E-19-616 (Dr. E. A. Starke) was supported by A-1319.

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	Dr. Starke
	E-19-616

EFFECTS OF INTERFACE PHENOMENA ON THE MECHANICAL BEHAVIOR OF METALS

by

Edgar A. Starke, Jr.
and
Billy R. Livesay

June, 1973

This research was supported by the Air Force Office of Scientific Research (AFSC) under Grant No. AFOSR-71-2064.

Qualified requestors may obtain additional copies from the Defense Documentation Center; all others should apply to the Clearinghouse for Federal Scientific and Technical Information.

SUMMARY OF RESEARCH

The mechanical behavior of crystalline materials is primarily explainable in terms of the dynamics of dislocations. A number of factors are known to influence dislocation dynamics in the neighborhood of an interface. The goals of this research program are to determine the relative importance of various interface parameters such as differential modulus, lattice misfit, crystallographic orientation, structure, etc. on the mechanical behavior of materials which contain interfaces, either on surfaces or internally.

Differential Modulus Effects

Our initial studies were concerned with the effect on mechanical behavior of a differential elastic modulus across an interface using single crystal composite specimens prepared in configurations which minimize the influence of other factors. Tensile specimens were prepared from thin copper single crystals and epitaxial metal films were grown on the surfaces by vapor deposition to provide desired interfaces. The principal material combinations studied were copper-nickel and copper-cobalt. In addition, copper films grown onto copper tensile specimens substrates were used to evaluate the influence of certain imperfections. The elastic modulus difference was found to provide the primary strengthening mechanism for Cu-Ni and Cu-Co interfaces. Surface and interfacial energy terms were also evaluated and found not significant compared to contributions from other interface factors. Crystal structure changes induced in cobalt coatings with deformation resulted in high work-hardening compared to that of the Cu-Ni systems. The major results of these studies have been published in Acta Metallurgica (1), and sixteen copies of this article are attached.

An interface model which more closely matches the modulus gradient expected for extended interfaces has been developed. The interface is partitioned into a sequence of discrete lamella to which specific elastic modulus values can be assigned. The number of lamella, their thickness and the individual modulus values are selected to approximate a desired interface configuration. The multiple image contributions to the force on a single screw dislocation parallel to the interface are then calculated by numerical summation procedures on the Georgia Tech Univac 1108 computer. A linearly decreasing, stepped variation of elastic modulus centered at the interface has been assumed. The systems for which these computations have been carried out correspond to Cu-Ni, Cu-Pt, Cu-Fe, Cu-Co and Cu-Re.

Composite specimens containing 30 to 50 layers have been prepared by the evaporation of alternate metal layers onto NaCl crystals. Individual layer thickness values were selected between 200 Å and 300 Å for all the specimens studied. Films of pure copper and of pure nickel were deposited so that each had a thickness corresponding to the accumulated Cu and Ni content of the composite structure. This allowed a direct comparison of the strength of films of the pure metals with that of the composite. The films were produced in the shape of our standard necked tensile specimens by depositing through photo-machined stainless steel masks onto the NaCl. The specimens were subsequently removed by floating them from the NaCl surface in distilled water. A strengthening effect of about 100% is found to occur for the composite specimens as compared to the combined strengths of the pure copper and pure nickel specimens.

The experiments mentioned above have been extended to include a new sample configuration and a different coating technique. The cylindrical sample design now being used permits sectioning along selected planes after tensile tests so that electron microscope examinations of the slip planes may be made.

This will allow direct observations of the dislocation-interface interactions. Interfaces produced by ion-plating onto single crystal substrates are being studied and compared with those produced previously by vapor deposition. Ion plated films are quite different from those produced by vapor deposition. The plating atoms strike the surface with significant energy to travel some distance into the material before coming to rest. This produces a film of unusually good adherence.

The penetration of the film atoms in vapor deposition is extremely small since they possess only thermal energy, thus the interface is quite sharp. In ion plating the penetration is significant with the result that there is a gradual interface which consists of a zone of substrate, sputtering and plating atoms. The physical status of this zone is unknown but may be any of a variety of forms from simply injected interstitial atoms to alloys of previously unobserved phases. The initial study in the ion plating effort is being directed to the characterization of this interface zone. The state and depth of the zone should be sensibly dependent on the sputtering and plating atoms, the accelerating voltage, and the temperature. Our first experiments will separate the parameters to determine their individual effects. We have done some preliminary sputtering experiments with argon gas and evaluated the strain using x-ray line broadening methods. The calculated strain was very small (3×10^{-4}), which may be real, but is likely due to the very thin zone relative to the x-ray penetration.

Mechanical properties such as critical resolved shear stress, extent of easy glide, and work hardening coefficient are being measured on the metal-metal single crystal systems which contain interfaces of various materials produced by ion plating. In addition, low cycle fatigue behavior is being studied since fatigue life is very sensitive to the surface condition. The deformation modes in both the primary and secondary slip systems of the substrate are being characterized by transmission electron microscopy and the interaction of the dislocations with the various interfaces are being examined.

Twin Interfaces in Single Crystals of Cu_3Au

Four flat (110) Cu_3Au single crystals were heat treated to various degrees of order; 0, 0.5, 0.8, and 1. These crystals were deformed at room temperature under plain-strain compression with the $[\bar{1}10]$ as the flow direction. A discontinuity, i.e., a sudden reduction, in work hardening rate was observed at certain points on the stress-strain curves. Concurrently, deformation bands were observed on the surface of the crystals. By comparison with optical micrographs of similar work on iron-cobalt alloys (2), it was concluded that these bands contained deformation twins. The stress levels associated with this deformation behavior increased rapidly with degree of order for high degrees of order; however, no such increase was noted when the degree of order was low. Assuming 1/6 $[11\bar{2}]$ (110) shear for mechanical twin formation for all degrees of order, changes in neighbor relationships during twinning are significant only for high degrees of order. The observed increase in twinning stress is attributed to this factor. The influence of stacking fault energy on the twinning stress was not as significant. However, a small decrease in twinning stress during the early stage of ordering is associated with a large decrease in stacking fault energy during this period.

The Effect of Interface Parameters on Friction and Wear Behavior

The surface of a bearing is subjected to complex combinations of mechanical stress modes. In the most simple cases, a volume of material near the surface alternately experiences stresses corresponding to tension, shear and compression. The usual situation is even more complicated with the introduction of other factors such as environmental effects and fatigue. The interface strengthening processes investigated in this program can be applied to the design of improved bearing materials using properly selected coating systems. A few simple experiments were carried out here to verify the validity of extending these concepts to friction and wear problems. A simple accelerated wear test was carried out on two copper crystals with regions coated with a 1500 Å Co-2.5% Fe film. A 25 micron diamond stylus was loaded normal to the flat specimen surface and the specimen was vibrated at 30 Hz with an amplitude of about 8mm. Wear tracks were made in both coated and uncoated regions of the crystals at loads ranging from 1-50 dyne and from 1800 to about 9000 cycles. SEM observations of the resulting wear tracks revealed that the cobalt alloy coating provided significantly greater wear resistance over that of the free surface.

Investigations of the Effect of Stable Crystal Structure of Coating on Mechanical Behavior

The Co-Fe alloys ranging from 0-6% Fe provide a useful system for investigations of the effect of the stable crystal structure of a coating on dislocation interactions at an interface since the stable structure of cobalt varies from hcp to double hcp to fcc in this alloy range. Preparation conditions identical to those employed for coatings of pure cobalt resulted in single crystal films for certain alloy compositions in this range. Mechanical tests conducted on copper crystals having 2000 Å coatings of alloys in this range have indicated the surprising result that a crystal specimen with a Co-2.5% Fe coating has a significantly lower yield stress than do crystals with either no coating or with the Co-6% Fe alloy coating. Analysis of the slip lines on low cycle fatigue specimens of copper crystals having these three surface configurations were consistent with the observed mechanical behavior.

Single slip systems were activated in crystals having either a free surface or coated with the Co-2.5% Fe alloy. The slip lines on the free surface were coarse whereas fine slip is found for a specimen coated with the Co-2.5% Fe alloy. A secondary slip system was activated with a Co-6% Fe coating indicating that the back stress of the 6% Fe alloy inhibited the operation of the primary slip system.

Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation

Four alloys based on the composition developed for 7050 by Alcoa for the Air Force have been prepared. These alloys include the commercial 7050 alloy, an alloy of identical composition with the exclusion of Zr, an alloy of identical composition with the exclusion of Zr and half of the Cu and an alloy of identical composition with the exclusion of Zr and all of the copper. This sequence of alloys will allow the isolation of various interface parameters, which is necessary for a theoretical analysis of the interaction of dislocations with the interfaces in an age hardening alloy such as 7050.

Our initial studies have been concerned with the kinetics of the decomposition of the super-saturated solid solid solution and the characterization of the microstructure developed. In addition techniques for growing single crystal, electron microscopy, small-angle x-ray scattering, and mechanical testing (both monotonic and cyclic) have been established.

Improvements in Facilities

A significant improvement was made in the Micromechanics Laboratory by rebuilding one of our devices into a highly versatile mechanical testing system. It is currently being used for the low cycle fatigue studies of 1-2mm thick coated copper crystals. Specimen mounting fixtures provide for tests either in air or in corrosion test cells. A schmidt trigger circuit and other electronic devices provide for completely automatic cycling and recording of data. Low-cycle fatigue facilities (a modified Instron Machine) have also been established for testing of bulk single and polycrystalline samples.

SCIENTIFIC PAPERS AND REPORTS

E. A. Starke, Jr., G. Kralik and V. Gerald, "Plasticity of Al-Ge Single Crystals Containing Small Fractions of Precipitates", Mater. Sci. and Engr. 11, 319-323 (1973).

B. R. Livesay and E. A Starke, Jr., "Dislocation Interactions at Interfaces", Acta Met. 21, 247-254 (1973).

B. R. Livesay and S. Spooner, "Magnetic Anisotropy of Ordered Cobalt Films", AIP Conference Proceedings No. 10, 1583 (1973).

Fu-Wen Ling and E. A. Starke, Jr., "Thermal Etching of β Ti-V Alloys", Metallography 5, 399-407 (1972).

D. A. Mauney, E. A. Starke, Jr. and R. F. Hochman, "Hydrogen Embrittlement and Stress Corrosion Cracking of Ti-Al Binary Alloys", Corrosion (in press).

K. C. Chen, Fu-Wen Ling and E. A. Starke, Jr., "Structure and Mechanical Properties of Stress-Ordered Ni_4Mo", Mater. Sci and Engr. (in press).

Fu-Wen Ling, H. J. Rack, and E. A. Starke, Jr., "Deformation of Metastable Beta Ti-V Alloys", Met. Trans. (in press).

Fu-Wen Ling, E. A. Starke, Jr. and B. G LeFevre, "Deformation Behavior of Beta Ti-V Alloys", (submitted to Met. Trans.).

PROFESSIONAL PERSONNEL

Dr. Edgar A. Starke, Jr., Professor of Metallurgy, School of Chemical Engineering;

Dr. Billy R. Livesay, Senior Research Scientist, Physical Sciences Division, Engineering Experiment Station.

GRADUATE STUDENTS

Mr. S. Chakrabortty, Ph.D. Candidate, "Twin Interfaces in Single Crystals of Cu_3Au"; Thesis Advisor: Professor E. A. Starke, Jr.

Mr. E. Y. Chen, Ph.D. Candidate, "Ion Plated Interfaces on Copper Single Crystals", Thesis Advisor: Professor E. A. Starke, Jr.

Mr. T. H. B. Sanders, Ph.D. Candidate, "Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation"; Thesis Advisor: Professor E. A. Starke, Jr.

Mr. A. Ansah, Ph.D. Candidate, "Effects of Microstructural Features on the Response of Aluminum Alloys to Cyclic Deformation"; Thesis Advisor: Professor E. A. Starke, Jr.

INTERACTIONS WITH OTHER GROUPS

Dr. E. A. Starke, Jr. attended the Spring Meeting of the Metallurgical Society in Boston in May, 1972. Attended Non-ferrous Metals Committee Meeting.

Dr. E. A. Starke, Jr. attended the Wire Association Meeting in Chicago in October, 1972 and received the Medal Award of the Nonferrous Division.

Dr. E. A. Starke,Jr. attended the Fall Meeting of the ASM-AIME in Cleveland in October, 1972 to meet with the Phys. Met. Committee to organize an Aluminum Symposium.

Dr. E. A. Starke, Jr. met with Drs. B. A. Wilcox of Battelle and H. L Gagel of AFML at AFML in December, 1972 to review abstracts and finalize program for Spring Meeting of AIME.

Dr. E. A. Starke, Jr. attended the symposium on Metallurgical Effects at High Strain Rates in Albuquerque in February, 1973.

Dr. E A. Starke, Jr. attended the Spring Meeting of AIME in Philadelphia in May, 1973 and was Chairman of a Session on the Advances in the Physical Metallurgy of Aluminum alloys.

Dr. E. A. Starke, Jr. was elected secretary of the Nonferrous Metals Committee of AIME and Chairman of a Committee to organize a symposium on modulated structures to be held in the Spring of 1974.

Dr. B. R. Livesay participated in a workshop for the preparation of a policy statement on deterioration of engineering materials for the National Commission on Materials Policy, June 26-28, 1972.

Dr. B. R. Livesay attended the 2nd National Conference on Crystal Growth, Princeton, July 30-Aug. 3, 1972.

Dr. B. R. Livesay attended 18th Annual Conference on Magnetism and Magnetic Materials, Denver, Colorado. He presented a paper there: "Magnetic Anisotropy of Oriented Cobalt Films".

Dr. B. R. Livesay visited Dr. I. R.Kramer at the Martin Marietta Corporation, Denver, Colorado and discussed common interests in surface mechanics.

Dr. B. R Livesay participated in an Air Force Materials Laboratory program in conjunction with the Ga. Tech School of Textile Engineering to determine the failure modes and wear resistance of certain fiber materials.

Dr. B. R. Livesay participated in an Army Missile Command program concerning the development and analysis of certain magnetic garnet materials for microwave applications.

PATENTABLE INVENTIONS

No patentable inventions have resulted from the sponsored research.

REFERENCES

1. B. R. Livesay and E. A. Starke, Jr., Acta Met. 21, 247 (1973).
2. G. Y. Chin, W. F. Hosford, and D. R. Mendorf, Proc. Ray. Soc. A. 309, 433(1969).

Respectfully submitted:

Dr. E. A. Starke, Jr.
Co-Principal Investigator

Dr. B. R. Livesay
Co-Principal Investigator

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13. ABSTRACT

The effect of various interface factors on the mechanical behavior of metals has been investigated. These include expitaial metal films on single crystal substrates, twin interfaces, and interfaces produced by the decomposition of super-saturated solid solutions. For the composite samples, elastic nodulus differences were found to provide the primary strengthening effect. Multilayered specimens, utilizing this strengthening effect, show an increase in yield strength of 100% over that expected for the combined strengths of the pure materials.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The purpose of the research was to examine in a fundamental manner the relative importance of various interface parameters on the mechanical behavior of materials. A broad research program included the study of such parameters as differential modulus, crystal structure, degree of order, stacking fault energy, type of interface, etc., and their effect on yield strength, work-hardening, low-cycle fatigue, and friction and wear. The material summarized in this report describes how the objectives of the program were achieved.

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EFFECTS OF INTERFACE PHENOMENA ON THE MECHANICAL BEHAVIOR OF METALS

by

Edgar A. Starke, Jr.
and
Billy R. Livesay

February, 1974

This research was supported by the Air Force Office of Scientific Research (AFSC) under Grant No. AFOSR-71-2064.

ABSTRACT

The purpose of the research was to examine in a fundamental manner the relative importance of various interface parameters on the mechanical behavior of materials. A broad research program included the study of such parameters as differential modulus, crystal structure, degree of order, stacking fault energy, type of interface, etc., and their effect on yield strength, workhardening, low-cycle fatigue, and friction and wear. The material summarized in this report describes how the objectives of the program were achieved.

SUMMARY OF RESEARCH

The mechanical behavior of structural materials is often critically dependent upon processes which occur at solid-solid, solid-liquid or at solid-gas interfaces. The direct interaction of an environmental gas or liquid with a metal surface is very short ranged due to electron screening. However, the environment can either introduce new solid-solid interfaces near the surface or else alter or even remove previously established interfaces. Significant long-ranged mechanical effects result from various dislocation processes which occur at or near solid-solid interfaces. The mechanical behavior of crystalline solids is primarily explainable in terms of the dynamics of dislocations in the neighborhood of an interface. The goals of this research program were to determine the relative importance of various interface parameters such as differential modulus, structure, etc. as well as types of interfaces such as stacking faults, antiphase domain boundaries, and twin boundaries on the mechanical behavior of materials which contain interfaces either on surfaces or internally. The results are briefly described below:

Differential Modulus Effects

The primary effort in the first part of this program was concerned with investigations of the effect on mechanical behavior of a differential modulus across an interface of single crystal composite samples prepared in configurations which minimized the influence of other factors. The elastic stress field resulting from the presence of a dislocation is long ranged and accounts for the interactions of dislocations with other dislocations and with such features as grain boundaries, precipitates, impurity atoms, surfaces and a variety of interfaces. Material discontinuities such as free surfaces or interfaces modify the stress field and there is a tendency for dislocation motion to lower the strain energy, e.g., a dislocation near a free surface will tend to move towards the surface in order to relieve the strain within the crystal.

Tensile specimens were prepared from thin copper single crystals and epitaxial metal films were grown on the surfaces by vapor deposition to provide desired interfaces. The principal material combinations studied were copper-nickel and copper-cobalt. In addition, copper films grown onto copper tensile specimen substrates were used to evaluate the influence of certain imperfections. The elastic modulus difference was found to provide the primary strengthening mechanism for Cu-Ni and Cu-Co interfaces. An abrupt interface, small lattice misfit and compatible crystal structures are required for this factor to play a dominant role. The role of other parameters such as interfacial impurities, crystallographic transistions in a coating material, crystallographic orientation and certain aspects of surface and interfacial energies were also evaluated.

The experiments mentioned above were extended to include a cylindrical sample configuration and a different coating technique. The cylindrical sample design permitted sectioning along selected planes after tensile tests so that electron microscope examinations of the slip planes could be made. This allowed direct observations of the dislocation-interface interactions. Interfaces produced by ion-plating onto single crystal substrates were studied and compared with those produced previously by vapor deposition. Ion plated films are quite different from those produced by vapor deposition. The plating atoms strike the surface with significant energy to travel some distance into the material before coming to rest. This produces a film of unusually good adherence.

The penetration of the film atoms in vapor deposition is extremely small since they possess only thermal energy, thus the interface is quite sharp. In ion plating the penetration is significant with the result that there is a gradual interface which consists of a zone of substrate, sputtering and plating atoms. The physical status of this zone is unknown but may be any of a variety of forms from simply injected interstitial atoms to alloys of previously unobserved phases. The initial study in the ion plating effort was directed to the characterization of this interface zone. The state and depth of the zone should be sensibly dependent on the sputtering and plating atoms, the accelerating voltage, and the temperature. Our first experiments were to separate the parameters to determine their individual effects. The strain produced in the surface of the copper single crystals during ion plating nickel films were measured using x-ray line broadening methods. The calculated strain was very small (3×10^{-4}), which may be real, but is likely due to the very thin zone relative to the x-ray penetration.

Mechanical properties such as critical resolved shear stress, extent of easy glide, and work hardening coefficient were measured on the metal-metal single crystal systems which contain interfaces of nickel and copper produced by ion plating. In addition, low cycle fatigue tests were conducted since fatigue life is very sensitive to the surface condition.

The nickel coated samples had superior mechanical properties when compared to uncoated and copper coated single crystals, emphasizing that the modulus effect is also very important for ion plated samples.

The calculations of dislocation interactions with layered coatings were based on an interface model consistent with the modulus gradient expected in extended interfaces. Treatments of dislocation interactions are usually carried out for either a single edge or screw dislocation in the neighborhood of an abrupt elastic modulus discontinuity. However, the model of an abrupt modulus discontinuity is not representative of a great many types of actual interface systems where interdiffusion occurs. Brief treatments of interface models involving a modulus gradient have been reported but these were essentially based on the resulting expression from calculations for an abrupt modulus discontinuity. In this work an interface model was constructed which more closely matched that expected in extended interfaces. The interface is partitioned into a sequence of discrete lamella to which specific elastic modulus values are assigned. The number of lamella, their thickness and the individual modulus values are selected to approximate a desired interface configuration. The multiple image contributions to the force on a single screw dislocation parallel to the interface are then calculated by numerical summation procedures on the Georgia Tech Univac 1108 computer. A linearly decreasing, stepped variation of elastic modulus centered at the interface was assumed. The systems for which these computations were carried out correspond to Cu-Ni, Cu-Pt, Cu-Fe, Cu-Co and Cu-Re. Forces on a dislocation as a function of the distance from the center of the interface were found to have functional behavior similar to that for single layered coatings. The equilibrium position (zero force) for a dislocation increased with the width of the diffusion region. However, the repulsive contribution of the interface at long range decreased with increasing interface width. This latter effect is much less significant for larger modulus differences, such as that for the Cu-Re interface.

It is proposed by J. S. Koehler that a composite specimen having alternate layers of materials with high and low elastic constants should have a greatly increased strength. Composite specimens of 30 to 50 layers were prepared here by evaporation of alternate metal layers onto the (100) cleavage plane of large sodium chloride crystals. This permitted unsupported tensile specimens of desired lamellar structures to be prepared for mechanical testing. The films were produced in the shape of our standard necked tensile specimens by depositing through photomachined stainless steel masks onto the NaCl. Individual layers were 200Å to 300Å thick. The specimens were subsequently removed by floating them from the NaCl surface in distilled water. A shutter arrangement in the vacuum deposition system was developed that permitted multiple tensile specimens to be deposited coincidentally for the two pure components and the composite structure. All the substrate crystals were located in a heater which maintained substrate temperatures at 300°C during deposition. The very difficult process of recovering specimens from the water and mounting them on tabs for tensile tests was greatly improved by developing a technique of coating the specimens with a soluble plastic film prior to removal from the sodium chloride. The strength of multiple layered Cu-Ni composite specimens were as much as 100% greater than the combined strengths of the corresponding pure copper and pure nickel specimens.

Influence of Surface Removal on the Mechanical Behavior of Copper Crystals

A series of experiments was conducted to investigate the effects of chemical removal of surface regions of coated and uncoated copper crystal tensile specimens. A coating may block dislocation motion by any of the several interface interaction processes mentioned earlier. A current topic of controversy concerns the relative importance of dislocation debris layers at the surface on surface-mechanical phenomena. Direct attempts to section deformed crystals in order to measure dislocation densities by transmission electron microscopy techniques are subject to questions concerning changes introduced by the sectioning and thinning operations. In this study, specimens were mounted on an electromagnetically loaded tensile testing machine. The machine was adapted with specimen mounting fixtures which permitted the tensile specimen to be emersed in liquid solutions during mechanical testing. The apparatus permits the specimen to freely deform without altering the applied load during a test. The liquid cell was used for both chemical and electro-chemical removal of specimen surfaces. The effect of chemical stripping of cobalt coatings was particularly significant. Single crystals with copper coatings experienced plastic strains between 0.5 and 1.4% due to only the chemical stripping of 1500Å pure cobalt films. These tests were conducted for tensile specimens loaded sufficiently to plastically deform the crystals about 0.2% prior to introducing the cobalt stripping solution. Similar tests were also conducted on copper polycrystal tensile specimens vacuum annealed for a day at 1000°C and coated with 1500Å cobalt. The anomalous strains induced by removing the coatings were usually less than 0.25%, but still significantly large. Much earlier attempts to observe coating effects on polycrystalline copper test specimens were not conclusive. For specimens stripped while unloaded, decreases in yield stress between 15-20% were observed upon reloading the specimen.

Interfaces in Single Crystals of Ordered and Disordered Cu_3Au

This effort was concerned with an experimental and theoretical analysis of the effect of ordering and the interfaces produced during the ordering reaction on the mechanical behavior of Cu_3Au. Two types of studies were performed. One was concerned with monotonic loading, and the deformation mode of primary interest was deformation twinning. The other study was concerned with cyclic loading and the deformation mode of primary interest was slip.

Mechanical Twinning takes place whenever imposed conditions make it difficult for the material to deform by slip alone. This difficulty increases with decreasing SFE since for materials with low SFE dynamic recovery by cross slip becomes difficult, and twinning becomes more favorable (1). Twinning also becomes favorable in this case because the separation of the partial dislocations, and thus the area of stacking faults increases with decreasing SFE.

Single crystals of Cu_3Au with various degrees of order were deformed under uniaxial tension, with their stress axis close to a <111> direction, at liquid nitrogen temperature and at room temperature. A <111> direction was chosen because earlier studies have shown that fcc metals twin at lower stresses when deformed in this direction (2) . Tensile tests were conducted in order to find the twinning stresses as a function of the degree of order for Cu_3Au.

Texture studies were made on Cu_3Au single crystals rolled both at room, and liquid nitrogen temperatures. These studies were used as a guide for choosing the deformation temperature for plane-strain compression studies. Cu_3Au single crystals with various degrees of order were deformed under plane-strain compression in order to study the texture development and to measure deformation twinning stresses. The compression plane of these crystals was (110) with the flow direction $[1\bar{1}0]$. Chin et al. (3) have shown that this is an easy orientation for deformation twinning to occur in fcc metals and alloys. Plane-strain compression studies were also conducted on a silver single crystal with the same orientation for comparison with Cu_3Au.

The results of the twinning study may be summarized as follows:

(1) Disordered and partially ordered crystals were found to twin by the mode described by $K_1 = (111)$, $K_2 = (11\bar{1})$, $\eta_1 = [11\bar{2}]$, $\eta_2 = [112]$ and $s = 0.707$. The shear stress required for this mode increases with the degree of order due to crystallographic restrictions imposed by the order. Completely ordered crystals were believed to twin by the mode described by $K_1 = (111)$, $K_2 = (001)$, $\eta_2 = [\bar{1}\bar{1}2]$, $\eta_2 = [110]$ and $s = 1.414$. The twinning stress required was comparatively low because ordering lowered the stacking fault energy and did not impose any crystallographic restrictions for this mode.

(2) The completely ordered Cu_3Au twins with audible clicks at an early stage of deformation under uniaxial tension, along a direction close

to <111>. Disordered and partially ordered Cu_3Au do not twin when deformed under uniaxial tension; but they do twin when deformed under (110) $[1\bar{1}0]$ plane-strain compression. The degree of long range order of the initially completely ordered Cu_3Au decreases with deformation, and twinning by the (111) $(11\bar{1})$ $[11\bar{2}]$ [112] 0.707 mode becomes possible by (110) $[1\bar{1}0]$ plane-strain compression.

(3) The only significant effect of twinning on the texture of Cu_3Au single crystals was found to be the rotation of the matrix due to the twinning shear.

Low cycle fatigue properties of fully ordered (3 specimens, strain amplitudes: $\pm$ 3.5%, $\pm$ 3.0%, $\pm$ 3.4%) and disordered (3 specimens, strain amplitudes: $\pm$ 3.5%, $\pm$ 3.0% $\pm$ 2.4%) Cu_3Au singles were determined. The disordered material exhibited continual cyclic strain hardening until cracking occurred. Rapid softening due to macro-crack propagation occurred at later stages of the fatigue process, which occupied only a small portion of total life. Fully ordered material exhibited an initial rapid cyclic strain hardening followed by cyclic strain softening. However, at higher strain amplitudes (> 3.0%), macrocracks were initiated in the early stages of the fatigue life, even before the material showed cyclic strain softening. For both ordered and disordered materials, kink bands developed on the surfaces in the early stages of the fatigue process. Macro-cracks were initiated at the boundary of the kink bands and propagated along the boundary. No stage II fatigue crack propagation has been observed. The proportion of the life spent in fatigue crack propagation is larger for the ordered material than for the disordered material.

The results of this study can be explained in terms of the degree of order and the effect of order on stacking faults and antiphase domain boundaries. The presence of these internal interfaces greatly influence the workhardening behavior of the single crystals.

The Effect of Interface Parameters on Friction and Wear Behavior

A limited series of experiments was conducted to examine the influence of metal interfaces on the mechanical deformation processes involved in unlubricated sliding contacts. The contacting surfaces in bearings are subjected to complex combinations of mechanical stress modes. In the most simple case, a volume of material near the surface alternately experiences stresses corresponding to tension, shear and compression. The usual situation is even more complicated with the introduction of other factors such as environmental effects and fatigue. Various coating combinations were prepared by growing layered structures on annealed copper crystal wafers having either (111) or (110) flat surfaces. All the tests were conducted in air. Frictional forces at low (1-15 dyne) and medium (100-200 dyne) stylus loads were measured using a low load friction measurement apparatus. This arrangement permitted the work hardening occuring during the "stick" portions of asperity interactions to be correlated with the crystallographic orientation of the crystal surface and the coating. The "stick" amplitudes were determined by the work hardening rate of the material near the crystal surface. Measurements on layered structures having Cu-Ni and Cu-Co interfaces showed that the work hardening rate at medium loads was increased with the introduction of an interface. A 25 micron stylus was used for these measurements. In addition, copper crystals were coated with composite layers so that Cu-Au, Cu-Ni-Au and Cu-Co-Au interface systems were formed with the external surface being gold. The low-load friction apparatus was arranged so that fine wire gold stylus could be employed and the electrical contact resistance monitored simultaneously with the mechanical forces. A direct correlation was found between work hardening rates of the surface layer and instantaneous values of the electrical contact resistance. A significantly lower and more stable sliding electrical contact was found for Au-Co-Cu composites than occured for a pure gold flat.

Accelerated wear tests were also conducted on copper crystals with and without 1500Å epitaxial coatings of Co-2.5%Fe. A 25 micron diamond stylus was loaded normal to the flat specimen surface and the specimen vibrated at 30 Hz with an amplitude of about 8 mm. Wear tracks were made in both coated and uncoated regions of the crystals at loads ranging from 1-50 dyne and from 1800 to about 9000 cycles. The cobalt alloy coating provided significantly greater wear resistance over that of the free surface.

Investigations of the Effect of Stable Crystal Structure of Coating on Mechanical Behavior

The effect of surface coatings on cyclical deformation was investigated for copper crystals having coatings of pure cobalt, Co-2.5%Fe and Co-6%Fe. The Co-Fe alloys ranging from 0-6% Fe provide a useful system for investigations of the effect of the stable crystal structure of a coating on dislocation interactions at an interface since the stable structure of cobalt varies from hcp to double hcp to fcc in this alloy range. Three point bend fatigue specimens were used in these studies. It was determined that the greatest effect of a surface coating occurred in the first few cycles of the fatigue hysteresis curve. After about 40 cycles, stress-strain curves and the work hardening rate per cycle appeared to be independent of the coating material. Initial stress-strain curves for specimens coated with the fcc Co-6%Fe alloy had both greater strength and work hardening per cycle than did the other combinations. This behavior is consistent with other reported investigations of deformation of hcp and fcc cobalt crystals which showed that fcc cobalt has a much higher work hardening rate than does hcp cobalt. Specimens in which the cobalt or cobalt alloy films were subsequently stripped after about 40 cycles were reduced in strength by at most 8%, and in some cases, not at all. Work-hardening rates did not change. Subsequent electropolishing did not appreciably influence the mechanical behavior of fatigue bend test specimens after 40 cycles indicating that surface dislocation debris does not represent a significant factor in the 40-90 cycle range.

Facilities

During the course of these investigations a versatile micro-mechanical testing system was developed to accommodate a number of types of thin specimen investigations. This apparatus provides greater precision as well as much greater flexibility for interface and environmental investigations than is available using the standard commercial testing machines which are primarily designed for large specimens. It employes a screw driven slide stage for specimen displacement and a number of different load cells provide force measurements ranging from less than 1 dyne to greater than 25 pounds. Specimen mounting fixtures provide for tests either in air or in corrosion test cells. New types of fixtures are easily adapted when a particular specimen test configuration is desired. A Schmidt trigger circuit and other electronic devices provide for completely automatic cycling for low cycle fatigue investigation and for recording of data. Other instruments, and sample preparation fixtures and techniques were also developed as needed during the course of this work.

Significant improvements in the sample preparation process used for thin tensile specimens were achieved by the construction of a special Bridgman type crystal grower. The tungsten resistive heating element, heat shielding and crystal mold were all enclosed in a vacuum chamber consisting of a 6 inch diameter pyrex cross. Multiple layered concentric radiation shields of molybdenum sheet surrounded the heating element. A water cooled copper cylinder at the interior wall of the chamber protected the pyrex from possibly dangerous temperature gradients. The hot zone, 1.5 inch diameter by 6 inch long, was defined at the lower end by molybdenum radiation shielding. Thermocouples along the lower end of the hot zone provided a temperature profile sufficiently accurate to predict the copper melt line to within 1/8 inch. This accuracy was particularly useful for using relatively small seed crystals.

The crystal mold was translated by a variac controlled motor drive system and a linear vacuum feed-thru. Split molds for various desired crystal shapes were machined from reactor grade graphite. Thin, flat strips of high purity copper were formed from stock cylinders by operating the crystal grower as a vacuum injection mold apparatus. The surface tension of liquid copper prevented the metal from flowing by gravity alone into a cavity smaller than about 2 mm thick. A graphite piston mold arrangement mounted in place of a crystal mold provided a convenient method for producing the thin copper blanks used for growing thin crystals. Although the apparatus has only been used for growing copper crystals, the materials used in the construction should also permit the growth of crystals from metals with melting points up to about 1500°C.

SCIENTIFIC PAPERS AND DISSERTATIONS

The following dissertations and papers resulted or will result from the support of this grant:

<u>Dissertations</u>

"Dislocation Interactions at Interfaces," Ph.D. dissertation for Billy R. Livesay, March, 1972.

"Deformation Modes of Copper-25 Atomic Percent Gold Alloy," Ph.D. dissertation for Saghana B. Chakrabortty, January, 1974.

"Effect of Ion Plating on the Low-Cycle Fatigue Behavior of Copper Single Crystals," Ph.D. dissertation for Edmund Y. Chen, in preparation.

<u>Papers</u>

B. R. Livesay and E. A. Starke, Jr., "Interaction of Dislocations With Interfaces," <u>Acta Met. 21</u> (1973) 247-254.

S. B. Chakrabortty and E. A. Starke, Jr., "Deformation Modes of Copper-25 Atomic Percent Gold Alloy," to be submitted to <u>Acta Met</u>.

E. Y. Chen and E. A. Starke, Jr., "Effects of Ion Plating on the Low-Cycle Fatigue Behavior of Copper Single Crystals," in preparation.

B. R. Livesay, "Mechanical Properties of Unsupported Multiple Layered Film Structures," in preparation.

B. R. Livesay, "Cobalt and Cobalt Alloy Coating Effects on Low-Cycle Fatigue of Copper Crystals," in preparation.

B. R. Livesay, "Investigations of Sliding Contact Behavior of Multiple Layered Coatings," in preparation.

B. R. Livesay, "A Versatile Micro-Mechanical Testing Machine," in preparation.

PERSONNEL

The following personnel participated in the research of this program:

Edgar A. Starke, Professor, Co-Project Director

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PATENTABLE INVENTIONS

No patentable inventions have resulted from the sponsored research.

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