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FINAL REPORT

submitted to:

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EQUIPMENT ACQUIRED BY

COLUMBIA UNIVERSITY

CENTER FOR STRATEGIC MATERIALS

JOHN K. TIEN

UNDER DOD UNIVERSITY RESEARCH INSTRUMENTATION PROGRAM

Grant Number: AFOSR-84-0225

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Columbia University purchased three pieces of equipment with grant money obtained from the Department of Defense under the University Research Instrumentation Program. This equipment includes

- 1) JSM-35cf Scanning Electron Microscope
- 2) PGT System4 Quantitative Energy Dispersive X-Ray Analysis Unit
- 3) Edwards E306A Coating/IBT 200 unit

For each item purchased there follows a brief summary describing the equipment, the manufacturer, and the cost. A status report on how well the equipment is operating in our labs will also be included. Finally, a summary of each program using the equipment donated by DOD is included with a description of how the equipment will help in attaining research objectives.

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH (AFSC)
NOTICE OF TRANSFER
This tech
approved
Distrib
MATTHEW J.
Chief, Technical Information Division

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Item 1 : JSM-35cf Scanning Electron Microscope

Manufacturer : JOEL USA, Inc.
11 Dearborn Road
Peabody, Massachusetts 01960

Consist of the following :

- A. Separate vibration isolated electron optical console, high vacuum system, power supply and connecting cables
- B. Camera (CSI) and Polaroid model 545 4x5 film holder
- C. Photo numbering unit with digital readout and print out of magnification, accelerating voltage, micron marker and exposure number (PNU-4)
- D. Built in air compressor for pneumatic valving
- E. Externally selectable X-Y adjustable multiple (3) aperture system for objective lens
- F. Capability for condenser and objective lens self maintaining column
- G. Focus Wobbler
- H. Dynamic focus correction for both specimen and CRT
- I. Automatic focus tracking
- J. Automatic magnification compensation for change of accelerating voltage and working distance
- k. Built in brightness and contrast meters
- L. Built in wave-form monitor and super rapid scan functions
- M. Y-modulation
- N. Eucentric goniometer stage with specimen and Z specimen movement (GSAZ)
- O. Gun Airlock (GIV)
- P. Ultra resolution photographic CRT (URH) (2000 lines guaranteed)
- Q. Multiple image (split screen) capability (MDD)
- R. Instant zoom device (dual magnification) (IZD)
- S. Image selector switch - 2 channel (IMS)
- T. Gamma control device with patented gamma monitor switch
- U. Scan Rotation and tilt correction device
- V. Backscatter electron detector (high sensitivity annular type)
- W. Installation, instruction, one set of operators manuals, recommended spare parts, one year warranty on all parts and labor and one year of emergency and periodic service
- Z. Water cooled Haskris Water Recirculator (115v), 35mm camera and adaptor, spare wehnelt assembly

Item 2 : PGT System 4 Energy Dispersive X-Ray
Analysis System

Manufacturer : Princeton Gamma-Tech
1200 State Road
Princeton, N.J. 08540

Consist of the following :

- A. Si(Li) detector
- B. Hardware Features: 64 K bytes DEC LSI 11/2 microcomputer, single floppy disk drive: double-sided dual density diskettes (1.2m Byte storage per disk), Winchester fixed hard disk with 10M Byte storage, 13" ultra high resolution color monitor, full typewriter style keyboard with special function keys, X-ray amplifier with pile up rejector, Analog to Digital Converter, Detector Bias Supply, Liquid Nitrogen Monitor, Enclosed cabinet on casters, 12 slot NIM Bin, 4 hardware window outputs for x-ray mapping
- C. 96 pre-defined element windows, user adjustable for integrals and mapping; simultaneous display of KLM marker lines for multiple elements with automatic peak element labeling; computer assisted element identification; unique computed display for spectral arithmetic; automatic computer-controlled energy calibration; simultaneous running of up to 14 windows for x-ray mapping
- D. Comparative Analysis Capability : Automatic Smooth, Scale, Ratio
- E. Background Subtraction
- F. Quantitative Analysis Capability: standardless analysis using Frame C overlap correction and includes ZAF matrix correction
- G. Automated Analysis Routines : user programmable sequencing; extended Beam/plot software
- H. Digital Beam Control: model 303B display and scan generator (including installation) ; digital color dot mapping
- I. Hard Copy Outputs : PGT Graphics printer/plotter includes serial I/O interface and spectral plotting software; Model 378 Polaroid Camera and hood
- J. Installation, introductory system training and one year warranty on all Princeton Gamma-Tech components

Item 3 : Edwards E306A Coating/IBT 200 unit

Manufacturer : Edwards High Vacuum Inc.
155-C New Boston Street
Woburn, Ma. 01801

Consists of the following :

- A. Edwards E306A basic coating unit
- B. H.T. Power supply
- C. Tripod
- D. Baffle plate
- E. Plasmaglo
- F. Filament holder
- G. Single carbon evaporation source
- H. Oil mist eliminator
- I. IBT 200 ion beam thinner
- J. Ion beam penetration switch
- K. IBT 200 process timer
- L. IBT 200 accesories include:
 - specimen holder;
 - cathode;
 - current probe;
 - specimen holder discs;
 - gun insulator

COST ANALYSIS

ITEM	COST
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1. JSM-35cf Scanning Electron Microscope	
2. PGT System 4 Quantitative Energy Dispersive	
X-Ray Analysis Unit	\$113,000
(Items 1 and 2 were purchased under the same vendor, JEOL, as a package. The price listed includes cost of both items 1 and 2).	
3. Edwards E306A Coating/IBT 200 unit	26,350
4. Transportation from Jeol	595
5. Transportation from Edwards	397

EQUIPMENT STATUS AND USE

The JSM-35cf Scanning Electron Microscope has been giving Columbia University quality performance. The sharp images due to an amazing resolution capability (5 nm) has allowed us to do quality microscopy which is an essential part of our research program. Not only is the JEOL microscope a quality instrument, but the service under our maintenance contract with JOEL has been superb. Although here at Columbia we provide daily maintenance for the electron microscope, every month or so during this start-up period, we have required a serviceman to make minor repairs or adjustments on the scope. JOEL has been prompt and efficient in meeting our needs.

The PGT System 4 Quantitative Energy Dispersive X-Ray Analysis Unit like the JOEL Scanning Electron Microscope has become an integral part of our microscopy arsenal. Qualitative and quantitative information on the chemistries of many materials have been obtained. The software is amazing. We give special praise to PGT for their dot mapping software, it has become very useful to many researchers in the materials science group. There has been no problem with the hardware. The only minor incident being that the belt on the floppy disk drive slipped off one day. In no time at all a serviceman came in and fixed it. We are satisfied with the PGT system and service.

The Edwards E306A Coating/IBT 200 unit has not been fully utilized yet. The ion beam thinning part of the Edwards did not work after we installed it. It has been returned to Edwards for repair under warranty. Edwards has

reported that repairs are progressing and the IBT should be returning soon.

The coating unit on the Edwards has been useful especially in preparing specimens for the SEM.

A description of the following DOD and government research and their use of the are included :

1. "Understanding the HIP Consolidation of P/M Nickel-Base Superalloys"(AFOSR)
2. "The Role of Cobalt in Nickel-Base Superalloys"(NASA)
3. "The Role of Molybdenum in Nickel-Base Superalloys"(ONR)
4. "Understanding Fibers/Matrix Compatability in TFRS Composites"(NASA)
5. "Directional Solidification and Creep/Fatigue Behavior of Nickel Aluminides"(ORNL-Martin Marietta-DOE)
6. "High Temperature Creep, Stress Rupture, and Crack Growth in Multi-phase Alloys"(NSF)

and other research of interest to DOD:

7. "Understanding the Comparative Fatigue Behavior of Cast and Wrought Superalloy in a low temperature Hot Corrosion Environment"(Special Metals/ Kelsey Hayes)
8. "Creep/Fatigue Interaction in Pb-Sn Solders"(IBM)
9. "Role of Niobium in Superalloys"(Niobium Products Corp.)

Principle investigator : John K. Tien

Project Name : "Understanding the HIP Consolidation of P/M
Nickel-Base Superalloys"

Project Number : AFOSR-82-0352A2

Summary of research work described in proposal:

To analytically model mechanisms and kinetics of the densification by HIP (Hot Isostatic Pressing) powder distribution, and to experimentally determine the effects on HIP kinetics and mechanisms of powder size distribution temperature and pressure. By understanding the operative HIP mechanisms, the conditions for HIP can be chosen in order to minimize the number of undeformed powder particles which leads the formation of undesirable PPB (prior particle boundaries).

Use of JEOL 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The SEM is used to analyze fracture surfaces of superplastically deformed specimens, to analyze and to examine the morphological microstructure of HIPed material, of superplastically deformed material, and of the original powders. An SEM is the only high resolution and high magnification tool that has the depth of field to image fine powders. The PGT System⁴ EDS is used to analyze PPB and grain boundaries in HIPed material.

Principle investigator : John K. Tien

Project Name : "The Role of Cobalt in Nickel-Base Superalloys"

Project Number : NASA NAG3-57

Summary of research work described in proposal:

To understand the role of cobalt in Nickel-base superalloys. The cobalt content in representative wrought nickel-base superalloys is systematically reduced from the standard amount with nickel substituting for the reduced cobalt content in the modified alloys. The effect of this process on both the physical metallurgy and mechanical properties of the alloy systems under study is then thoroughly investigated. Current research is aimed more at alloy processing. An attempt is being made at processing a P/M superalloy as a cast/wrought alloy by applying the knowledge we have already gained on the effect of cobalt on cast/wrought alloys through previous years of research.

Use of JEOL 35cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The JEOL 35cf SM and the PGT System⁴ EDS units are being used to study the phases present in an as-cast ingot of a high strength superalloy as well as the relationships between and changes in these phases after exposing the alloy to various heat treatments. Fracture surfaces are studied with the JEOL 35cf.

Principle investigator : John K. Tien

Project Name : "The Role of Molybdenum in Nickel-Base Superalloys"

Project Number : N00014-83-k-0223

Summary of research work described in proposal:

To determine the effects of substituting refractory element Mo for other strategic refractory elements W, Nb, and Ta in three commercial superalloys, U-710, B-1900 and IN-738 . The changes in microstructure, chemistries, mechanical properties and hot corrosion behavior will be studied.

Use of JEOL 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The JEOL 35 cf SM will be used to characterize microstructures, to analyze fractured surfaces of mechanically tested material , and to profile the hot corrosion behavior of the modified alloys. The PGT System⁴ EDS unit will be used for phase identification and chemical analysis of the cast alloys, and the Edwards IBT/Vacuum coater unit will be used to make foils for TEM observation as well as to prepare specimens for SEM imaging.

Principle investigator : John K. Tien

Project Name : "Understanding Fibers/Matrix Compatability in TFRS
Composites"

Project Number : NASA-NAG3-410

Summary of research work described in proposal:

To determine the nature, kinetics and extent of Fiber/Matrix reactions and/or microstructural changes that occur in Tungsten fiber reinforced superalloys after prolonged exposure at high temperatures. The base matrix for this study is Incoloy 903, the fibers are tungsten doped with 15% ThO₂ and the temperature range is 1000 C to 1200 C.

Use of JEOL 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The PGT System⁴ EDS unit in conjunction with the JEOL 35cf SEM is used to obtain the as annealed chemistry profiles over the entire composite diffusion couples and to metallographically image these diffusion couples. The Edwards IBT/Vacuum coater will be used to obtain thin foils of the composites which is an otherwise impossible task.

Principle investigator : John K. Tien

Project Name : "Directional Solidification and Creep/Fatigue
Behavior of Nickel Aluminides"

Project Number : Mrtta 19X-89664C

Summary of research work described in proposal:

The parameters affecting solidification and creep/fatigue behavior of directionally solidified Ni₃Al (with boron and titanium alloying additions) are being investigated. The growth rate is being varied to determine the effect on microstructure, porosity, segregation and mechanical properties. Hot Isostatic Pressure (HIP) studies will determine the pressure and temperature where pore closure occurs without deviatoric yielding and subsequent recrystallization. High temperature stress controlled fatigue tests will determine the amount of creep/fatigue interaction in single crystal and directionally solidified Ni₃Al.

Use JEOL 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit"

The microstructure and especially the porosity of directionally solidified and monocrystalline nickel aluminide alloy is being examined using the JOEL 35cf SM. Segregation analysis will be performed using the PGT System⁴ EDS along with the SEM backscatter electron imaging mode. Thin foils of mechanically tested Ni₃Al prepared on the Edwards IBT unit will undergo TEM deformation analysis. Fractographic analysis will be performed to determine the extent of creep/fatigue interaction using the JEOL 35 cf SM.

Principle investigator : John K. Tien

Project Name : "High Temperature Creep, Stress Rupture, and Crack
Growth in Multi-phase alloys"

Project Number : DMR-80-11402

Summary of research work described in proposal:

Recently Columbia University has documented a correlation between primary creep, steady state creep and stress rupture for two fine grain polycrystalline superalloys. It is the purpose of this investigation to document this correlation for variations in grain size and in gamma prime morphology. Also of optimal importance this correlation must be documented for both Haper Dorn and power law creep regimes for a wrought polycrystalline superalloy.

Use of JEOL 35 cf SM, PGT System4 EDS, or Edwards IBT/Vacuum coater unit:

The JEOL 35 cf SM will be utilized to document all of the fracture surfaces for failure mode of each of the creep and creep/fatigue tests. The PGT/EDS will be utilized for matrix composition sampling and for observation and sampling of grain boundary carbides.

Principle investigator : John K. Tien

Project Name : "Understanding the Comparative Fatigue Behavior of Cast and Wrought Superalloy in a Low Temperature Hot Corrosion Environment"

Summary of research work described in proposal:

To determine whether the fatigue curves in air and in sulfate/chloride film(salt) diverge and remain divergent or they diverge and then converge at very high cycles and low mean stresses. The alloys studied are IN-738 and U-720 at 1300F and 5Hz. Constant maximum stress tests will be used to map fatigue initiation and propagation.

Use of Jeol 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The Jeol 35 cf SM is used to characterize and investigate the specimen fracture surfaces including the morphology and local chemistry of the initiation sites. Both the SEI and BEI modes are used to investigate oxide film and salt initiated fatigue cracks along the gage length. The PGT System⁴ EDS has been used to characterize initiating carbides and inclusions and to investigate chemical variations near crack tips.

Principle investigator : John K. Tien

Project Name : "Creep/Fatigue Interaction in Pb-Sn Solders"

Project Number : IBM-440214

Summary of research work described in proposal:

PbSn solders, used extensively in the electronics industry, are subject to operating conditions which can be best simulated through creep-fatigue testing. The PbSn eutectic, and 81Pb-19Zn are subject to creep, cyclic creep and tensile loading conditions at 25C in an effort to determine the effect of load cycling frequency, mean stress, and volume fraction of Pb-rich phase on minimum creep rate and rupture life.

Use of Jeol 35 cf SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The SEM will be used in both secondary and backscatter electron imaging modes to characterize the eutectic PbSn microstructure, i.e determining interlamellar spacing. In addition, each fractured specimen is observed and photographed using the 35mm camera in the secondary electron imaging mode (SEI). The PGT EDS is used to determine exact composition and presence of impurities which may have affected mechanical properties.

Principle investigator : John K. Tien

Project Name : "Role of Niobium in Superalloys"

Summary of research work described in proposal:

This program will determine TTT diagrams for 10 modified IN718 alloys. In these diagrams three phases are of primary importance. These phases are the gamma-prime phase, the gamma-double prime phase, and the delta phase. The latter phase is detrimental to the alloy and our efforts are directed towards suppressing the precipitation of the delta phase.

Use of Jeol 35 of SM, PGT System⁴ EDS, or Edwards IBT/Vacuum coater unit:

The JSM-35 of scanning microscope is instrumental in identifying and analyzing the delta phase in specimens heat treated at different times and temperatures. The backscatter image mode intensifies topographical variations along the specimen surface which has been crucial in pinpointing the delta phase. The PGT System⁴ EDS unit aids in identifying the delta phase through chemical analysis. Dot mapping with the PGT allows for a quick chemical survey of a large area and has saved many filament hours. The Edwards coating system will be used to carbon coat specimens to enhance the resolution of the SEM image.

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