

The Institute of Research

LEHIGH UNIVERSITY



Office of Grants and Research Contracts
Attention: Code SC
TO: National Aeronautics and Space Administration
Washington, D. C. 20546

SUBJECT: Second Semi-annual Progress Report
on
"Investigation of the Solidification, Structure and Properties
of Eutectic Alloys Including Consideration of Properties Control"
NASA Research Grant: NGR-39-007-007

GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 1.00

Microfiche (MF) 1.50

853 July 65

SUBMITTED BY:

R. W. Hertzberg
R. W. Hertzberg, Assistant Professor
Co-investigator

July 14, 1966

DATE:

R. W. Kraft
R. W. Kraft, Professor
Principal Investigator

FACILITY FORM 802

N66 32264

(ACCESSION NUMBER)

17

(PAGES)

CR-76668

(NASA CR OR TMX OR AD NUMBER)

(THRU)

1

(CODE)

17

(CATEGORY)

INTRODUCTION

During this past report period, a renewal proposal to the present NGR-39-007-007 on unidirectional solidification of eutectic alloys was accepted. It was proposed that work would continue in the subject areas undertaken in the first grant period and, in addition, be initiated on the thermomagnetic and thermoelectric properties of controlled eutectic alloys. Since few studies have been made of the physical properties of unidirectionally solidified eutectic alloys at Lehigh and elsewhere, such an effort represents a significant departure from present research activities. The addition of Dr. Richard Tauber to the staff of the Metallurgy and Materials Science Department and the Materials Research Center has strengthened our ability to make significant progress in this area. A brief description of Dr. Tauber's background may be found at the end of this report.

The bulk of this report consists of brief statements of the objectives, plans and status of each of the several separate but related research programs currently being investigated on this grant by the graduate research assistants and senior staff men associated with the work.

RESEARCH IN PROGRESS

Microstructural Stability at Elevated Temperatures

Before the effect of temperature and stress on the microstructural stability of a fibrous Al-NiAl₃ eutectic can be satisfactorily evaluated, it is necessary that a model be developed by employing essentially the same procedure as used by L. D. Graham for the lamellar Cu-CuAl₂ eutectic (i.e., effects of temperature without stress) (1). This model would then serve as a foundation on which to base the additional effects of an applied stress on microstructural stability.

The first phase of this work consists of heat treating specimens controlled at growth rates of 2, 8, and 32 cm/hr. for times of 10, 100, and 500 hrs., at

temperatures of 590, 610, and 630°C. Difficulties were encountered, however, for it was found that at growth rates above 7 cm/hr. a pronounced eutectic colony microstructure resulted, probably caused by impurity segregation. To achieve the high purity needed at these high growth rates, an ingot approximately 1/2 inch in diameter by 22 inches long was zone refined using an RF induction unit with a cooling coil. Ten passes at 8 cm/hr. and a final pass at 20 cm/hr. were made. Transverse metallographic sections revealed the first 3/4 of the ingot to be aligned, indicating that most of the impurities were pushed to the tail-end of the ingot.

The ingot was then cut into three equal lengths and two specimens controlled horizontally at growth rates of 2 and 8 cm/hr. These have been sectioned into wafers 3/16 inch in thickness and are ready for subsequent heat treatment. The third specimen sectioned from the head-end and presumably of highest purity is to be controlled in a vertical, water-cooled end quench apparatus at 32 cm/hr.

During this period replication techniques were developed for observation of the microstructure in the electron microscope.

In the next report period, it is anticipated that most of the non-stress thermal stability studies will have been completed.

Elevated Temperature Materials

During this past report period, considerable progress has been made in the study of the Ni-Ni₃Nb eutectic alloy system. The published eutectic composition, reported to be in error in the first progress report, has been established at 22.65 weight per cent Nb. Several ingots of this composition have been prepared and unidirectionally solidified at about 4.5 cm/hr. Metallographic sections, both longitudinal and transverse to the growth direction, reveal the structure to be lamellar with alternating platelets of the Ni and Ni₃Nb phase (Figures 1 and 2).

Since the volume fraction of Ni is 78%, the larger platelets may be identified as the Ni phase. Based upon the encouraging results of the solidification

and microstructural studies, a 20-lb. master heat has been vacuum melted and cast into an investment mold producing bars suitable for subsequent processing. Thermal shock of the refractory boats has temporarily delayed production of unidirectionally solidified specimens. A superior crucible has been ordered and shipment is expected shortly so that specimen preparation may be accelerated.

While ingots containing controlled microstructure made to date have been too small for preparation of tensile or creep specimens, some preliminary studies of the mechanical behavior of this alloy have been made. One such ingot was deliberately bent in a bench vise to obtain a qualitative measure of the degree of ductility that might be expected from the Ni-Ni₃Nb controlled microstructure. Figure 3 offers evidence of considerable ductility in the alloy. Another ingot, metallographically polished and etched prior to deformation, revealed slip line penetration in both Ni and Ni₃Nb phases. This type of investigation will be actively pursued in the next report period.

Work on the Co-Co₂Nb eutectic system has temporarily been discontinued since the first report period. The allotropic phase transformation of cobalt and a reported ordering effect in the system have discouraged further work. Emphasis has, therefore, been shifted to the Ni-Ni₃Nb system which was discussed above.

To prepare for the elevated temperature mechanical test program which should begin in the next reporting period, necessary equipment has been obtained. Two Riehle 12,000-pound creep rupture stands with temperature controllers donated to Lehigh University by the E. I. duPont Company and four 1500-pound capacity Dennison units will be used in this investigation. Temperature controllers have been purchased for the Dennison units and all creep units fitted with 1800°F Marshall creep furnaces. These latter items of equipment were all purchased on this grant, as budgeted.

Mechanical Properties of Al-CuAl₂ Eutectic

Previous work by the principal and co-investigators revealed the structure of the Al-CuAl₂ eutectic to be lamellar and the bend strength strongly dependent upon the angle between the lamellae orientation and flexural stress direction (2,3). In a recent paper, Salkind et al (4) discussed the potential of lamellar-type eutectics and concluded that they were inferior to eutectic systems exhibiting a rod-like morphology. This conclusion was based mainly on a calculation of the theoretical strength that would be expected from the Al-CuAl₂ system. Using the modulus of elasticity values obtained from flexural tests for the transverse direction (3) and elastic strain measurements of tension specimens aligned parallel to the growth direction, they found the expected theoretical strength to be almost twice that of experimentally determined values (i.e., 75,000 psi vs. 35,000-43,000 psi).

Though it was not the original intent of this research project to study the mechanical behavior of this system, it was felt that use of transverse elastic modulus data in the calculation of longitudinal strength was suspect. Therefore, an exploratory program to determine the longitudinal elastic properties in the Al-CuAl₂ system was begun. To summarize these results (to be discussed at length in future publications), it was found that the elastic modulus of the CuAl₂ phase was considerably less than that reported in transverse directions. Using the newly determined modulus value in the strength calculation, the theoretical strength of the Al-CuAl₂ eutectic system was found to be about 41,500 psi, in excellent agreement with experimentally determined tensile strength. From these results, it is not possible to state that rod-type eutectic microstructures are any better than lamellar type. In fact, lamellar eutectics may prove to be more desirable since the volume fraction of the strong intermetallic phase is larger and the microstructure capable of supporting a

bi-axial stress distribution.

In view of the above results, a decision was made to continue work on the Al-CuAl₂ system. Both flexural and compression test programs have been initiated to investigate the effect of lamellar size on composite strength and the deformation and fracture modes during compression testing. Ingots have been unidirectionally solidified at growth rates of 0.6, 1.0, 1.9 and 4.0 cm/hr. Four-point loading flexural test results obtained to date revealed no apparent change in modulus of rupture data for specimens from ingots grown at the three slowest rates, while a definite increase in strength was noted for the specimens prepared at 4.0 cm/hr. Due to the limited ductility exhibited by this alloy system, considerable scatter in the results may be expected so more testing is needed before conclusions may be drawn.

Compression tests parallel to the ingot growth direction have yielded ultimate strengths in excess of 60,000 psi. This marked increase in strength under totally compressive loading is reasonable in light of the low tensile ductility exhibited by the alloy. Post-fracture metallographic examination revealed striking deformation modes; lamellae buckling and kink band formation were readily observed during room temperature tests (Fig. 4). Deformation kinking had previously only been observed during elevated temperature deformation studies (5). Close examination of the kinked area gave evidence of CuAl₂ ductility since most of the lamellae were not visibly fractured (Fig. 5). In addition to the kink bands, there were many parallel lines running almost perpendicularly to the kinks and almost parallel to the lamellae. The cause of these markings is not known, nor is it clear why kinking is not observed in all grains. These questions will be actively pursued in the next report period.

Fatigue Studies of Al-Al₃Ni Eutectic

Satisfactory progress has been made in the investigation of the fatigue

behavior of rod-like Al-Al₃Ni. It had originally been decided to investigate fatigue behavior by constant strain tests with the eutectic specimen bonded to a high strength cantilever beam. To this end, material was purchased and two cantilever beams with tapered width sections (to produce regions of constant bending moment) fabricated. While the cantilever beams were being readied for use in the test program, several ingots were machined into notched tensile specimens to study the notch sensitivity of the Al-Al₃Ni eutectic in the as-cast and unidirectionally solidified condition. In addition, constant load range fatigue tests on both microstructural forms were planned. Preliminary results reveal that the controlled structure is extremely notch-sensitive. A V-notch with 50% reduction of area lowered the tensile strength level to 30,000 psi from 45,000 psi in the unnotched condition. This considerable amount of notch sensitivity raises concern about potential use of this class of alloys and reinforced composites in general. These experiments will be continued along with notch testing of the as-cast material which may not display the same degree of notch brittle behavior.

Two controlled specimens have been fatigue tested to failure at stress ranges of 16,300 psi and 13,300 psi and failed after 278 and 1712 cycles, respectively. A most interesting observation was that shear failure, 20 to 30 degrees from the tensile axis, accompanied fracture of all monotonically loaded test bars and the 278 cycle life specimen, while almost flat fracture occurred in the 1712 cycle life specimen. This fracture mode transition will be examined carefully in the next report period, since it may hold an important clue to fatigue behavior of composite materials.

The active program of notched bar tensile and fatigue work on the Al-Al₃Ni system and the new program on the Al-CuAl₂ alloy has restricted activity relating to the constant strain tests. It is hoped that further progress will be made in this program in the next report period.

Physical Property Measurements

Due to the alignment of controlled eutectic microstructures, considerable anisotropy of physical properties may be expected. Recently completed Ph.D. theses by Albright (6) and Levy (7) at Lehigh University of unidirectionally solidified eutectic alloys have revealed considerable anisotropy of magnetic and superconducting properties, respectively. Based upon this evidence, it is reasonable to assume that thermomagnetic and thermoelectric properties shall also be highly directional. In fact, it has been shown (8) that the thermomagnetic figure of merit is enhanced by the addition of a controlled rod-like second phase in a semiconducting matrix.

The eutectic alloy chosen to initiate this investigation was the Bi-Zn system since the Bi matrix phase has a high figure of merit. The eutectic reaction occurs at 2.7 w/o Zn at 254.5°C. Specimens were prepared by encapsulating 99.999% Bi and Zn in Vycor capsules and melting the charge. Preliminary attempts to align the microstructure by unidirectional solidification at 0.64 and 1.0 cm/hr. have been successful. Metallographic examination, using both chemical and electrolytic preparation techniques, reveals the microstructure to contain Zn platelets aligned parallel to the growth direction within a continuous Bi matrix.

Electrical resistivity was measured on a controlled Bi-Zn specimen, both perpendicular and parallel to the platelet orientation and found to be 3.385×10^{-4} ohm-cm and 1.635×10^{-4} ohm-cm, respectively. An apparatus designed to measure transport coefficients such as electrical and thermal conductivity and Seebeck, Nernst, Ettinghausen and Hall coefficients has been built. A special dewar arrangement and a specimen holder has been designed and fabricated to permit measurement of the various coefficients in the temperature range of 77°K to 300°K. This work will be initiated in the next reporting period. A six-inch Varian adjustable magnet with field control for the above apparatus has been ordered and shipment expected shortly.

PERSONNEL

As mentioned above, Dr. Richard Tauber, Assistant Professor of Metallurgy and Materials Science, is actively participating in the new research effort on the physical properties of controlled eutectic microstructures effective 15 June 1966. Dr. Tauber joined the staff at Lehigh in Feb. 1966 after completing his Ph.D. work at N.Y.U. on the optical and electrical properties of PbTe-SnTe alloys. Papers based upon his dissertation research are in press. His field of specialty is physical properties, especially of thermoelectric semiconducting materials. In addition to his work on eutectic alloy systems, Dr. Tauber is initiating other research on multicomponent single-phase thermoelectric materials to follow-up on his Ph.D. dissertation research. Assisting Dr. Tauber in this research effort will be Mr. Sol Musikant who had been initially assisting Dr. Hertzberg.

The principal investigator and co-investigator are also most happy to report that a student of outstanding potential has been added to the research program. Mr. William Hoover, an honor graduate of Lafayette College with a degree in metallurgical engineering, has been elected to both Phi Beta Kappa and Tau Beta Pi, honorary fraternities. Joining the staff June 15, Mr. Hoover will initially assist Dr. Hertzberg and eventually begin his own research program on the mechanical behavior of controlled eutectics.

With these additions, the total staff now working on research under this grant is as follows:

<u>Name</u>	<u>Capacity</u>	<u>Percentage Participation</u>	
		<u>Summer '66</u>	<u>Academic Year</u>
Prof. R. W. Kraft	Principal Investigator	25%	10%
Asst. Prof. R.W. Hertzberg	Co-investigator	50%	25%
Asst. Prof. R. Tauber	Sr. Res. Associate	40%	20%

Cdr. T. Quinn	Grad. Res. Asst.	100%*	50%*
Mr. W.G. Watson	Grad. Res. Asst.	100%	50%
Mr. S. Musikant	Grad. Res. Asst.	100%	50%
Mr. W. Hoover	Grad. Res. Asst.	100%	50%

* As noted in a previous report Cdr. Quinn's research expenses are being born by this grant but he is drawing no stipend.

REFERENCES

- 1). L.D. Graham and R.W. Kraft, AIME Trans 236 No. 1, 1966, p. 94.
- 2). R.W. Kraft and D.L. Albright, Trans AIME 221 (1966), p.95.
- 3). R.W. Hertzberg, F.D. Lemkey, and J.A. Ford, Trans AIME 233 (1965) p.342.
- 4). M.J. Salking, F.D. George, F.D. Lemkey, B.J. Bayles and J.A. Ford, Chemical Engineering Progress Vol 62, No. 3 March 1966, p. 52.
- 5). A. Yue and W. Coons, metallographic exhibition, ASM Convention, Philidelphia October, 1964.
- 6). D.A. Albright, Ph.D. Thesis, Lehigh University, September, 1965.
- 7). S. Levy, Ph. D. Thesis , Lehigh University, June, 1965.
- 8). H. Wagini and H. Weiss, Solid State Electronics, 8 ,241, (1965).



Fig. 1 Longitudinal
section of controlled
Ni-Ni₃Nb eutectic
250X.

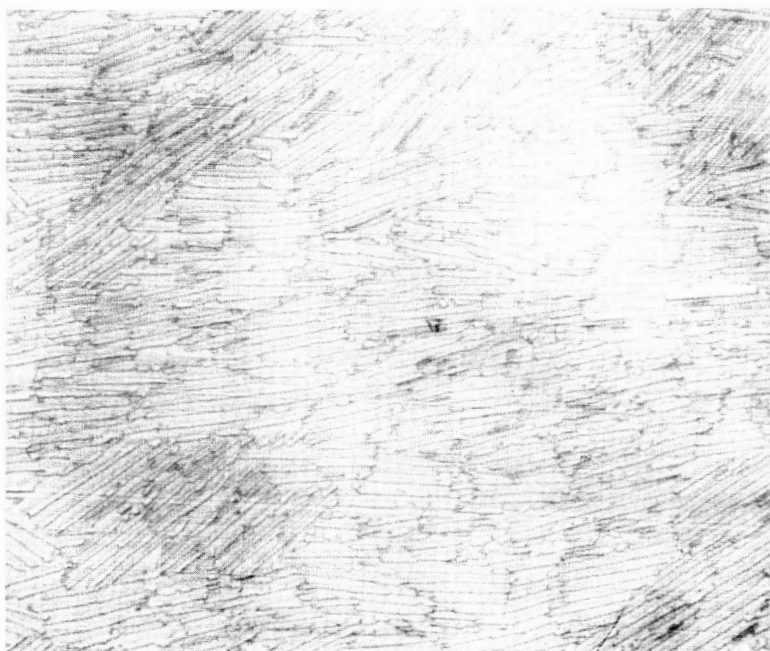


Fig. 2 Transverse
section of controlled
Ni-Ni₃Nb eutectic
250X.

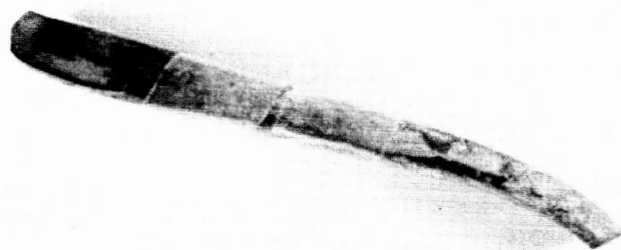


Fig. 3 Evidence of
deformability of
controlled Ni-Ni₃Nb
ingot.

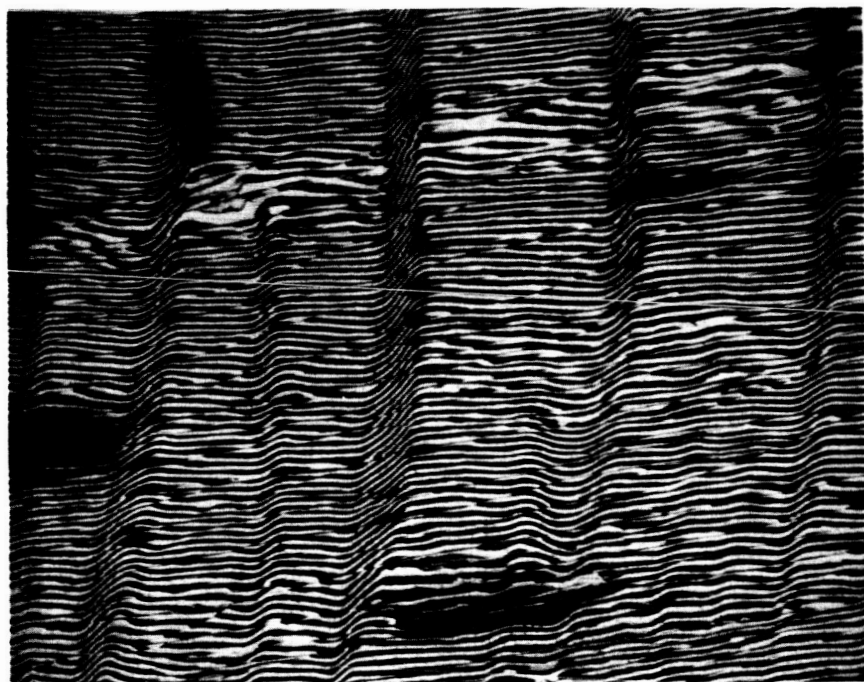


Fig. 4. Evidence of Kink-Band Formation in Al-CuAl₂ Resulting From Compressive Loading. 100X



Figure 5. Kink- Band in Al-CuAl₂ with some unbroken CuAl₂ Platelets. 500X