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INVESTIGATION OF THE SOLIDIFICATION, STRUCTURE AND PROPERTIES OF EUTECTIC ALLOYS

INTRODUCTION:

The research being done under Grant NGR 39-007-007 consists of several distinct, but related, projects on 'controlled eutectics'. These are highly anisotropic binary eutectic alloys produced by unidirectional solidification under closely controlled conditions. Although controlled eutectics have been under investigation for over six years in various laboratories, each of the several projects being studied on this grant was initiated within the last year and a half (in one case, less than a year ago) when grant funds and graduate research assistants became available. Each program is now well under way and substantial progress has been made although none of the projects have yet been pursued to the point where complete technical papers can be prepared. It is anticipated that at least one of the projects and perhaps one or two others will have reached this stage in the next six months. The others will require more effort and a renewal proposal for continuation of the grant into the third year is being prepared with this in mind.

The sections which follow describe the work in progress according to the following scheme.

Ambient Temperature Fatigue and Fracture Studies

Elevated Temperature Programs

Physical Properties

AMBIENT TEMPERATURE FATIGUE AND FRACTURE STUDIES:

One of the main objectives of this research program is to study the nature of a dynamic stress state on the behavior of composite materials. The Al-Al_3Ni eutectic system was chosen for this investigation since it has proven to possess mechanical reinforcing capability when the two phases are aligned parallel to the intended stress direction. One primary concern in this investigation was a study of the effect of cyclical stresses upon the integrity of the fiber-matrix interface. While interface strength might be sufficient for monotonic loading conditions serious deterioration of the interface strength could result from the accumulation of damage due to reversed loading.

Al-Al_3Ni Fatigue Studies: The initial testing for this program was conducted on specimens which were unidirectionally solidified on a horizontal zone refiner. Test results were found to be unacceptable because the Al_3Ni fibers were not parallel to the longitudinal axis of the specimen resulting in tensile strengths of only about 60% of the expected values. Consequently, it was decided that all specimens for this program would be grown on a vertical zone refiner.

Five ingots of high purity Al-Al_3Ni have been unidirectionally solidified on a vertical zone refiner at a growth rate of 6 cm/hr. These were machined into 0.25 inch diameter notched tensile specimens with a one inch gage length. The notch reduced the diameter to 0.188 inches with a notch root radius of less than .002 inch. (A decision was made to utilize a notched-round type specimen for the low cycle fatigue tests instead of a smooth specimen. This was done to assure uniformity and predictability of the fracture initiation site since most smooth specimens tend to fail near the shoulder area in tension testing.) These samples were then tested in tension-tension fatigue using an Instron Universal Testing Machine. The minimum net stress was maintained at 2,000 psi while the maximum stress was varied from 19,000 to 28,000 psi. During testing, total strain to failure was measured over the one inch gage length containing the notch. These values will be later compared with maximum strain measured from static tests for evidence of plastic strain accumulation. Table I summarizes the fatigue data obtained thus far in the test program. After testing, the fracture surfaces were replicated for fractographic studies and then Ni plated and sectioned for metallographic examination.

TABLE I

Fatigue Data of $Al\text{-}Al_3Ni$

Specimen	σ max, psi	Cycles to failure	Total Strain
I1	29,800	8	-
I2	26,000	41	0.87%
I3	20,000	268	1.16%
I4	18,200	1054	-
I5	16,880	1444	1.45%

The initial metallographic and fractographic work indicated that, in fatigue, the crack propagated at or near the fiber-matrix interface to a much larger degree than in static tension. Figure 1 is a photomicrograph of a sectioned specimen showing a typical tensile failure resulting from fiber fracture. Note that there is no clear evidence of failure at or near the fiber-matrix interface. However, Figure 2 shows a fatigue fracture with evidence of failure at or near the interface along with fiber failure found predominantly in tensile fracture. Fractographic observations were in agreement with the above findings. Figure 3 depicts a transition region from "dimpled" rupture produced by fiber fracture to flat fracture with longitudinal profiles of Al_3Ni fibers caused by interfacial or near interfacial decohesion. Figure 4 reveals greater detail of the Al_3Ni fibers found on the fracture surface. It is not clear at this time whether the ratchet-like markings found predominantly along the length of the fibers are produced by the actual fracture process or are a result of rubbing together of the two fractured surfaces.

These preliminary findings suggest that the cyclic action of fatigue loading causes damage at or near the fiber-matrix interface. It is felt that the fatigue crack then propagates through this damaged area. The measurements of total strain to failure show that as the number of cycles to failure increases, the total strain to failure increases also (Table I). The significance of this observation will be considered in the next report period when more data are obtained.

At the present time, more specimens are being tested with anticipated fatigue lives of up to 5,000 cycles to provide verification of the S-N curve and the total strain to failure vs. number of cycles relationship. In addition, this work will more firmly establish the failure mechanism in the low cycle region.

Preparations are also being made to test in a range of 5,000 to 10^6 cycles on a Baldwin high speed fatigue machine. Since the specimens for this machine are of a different shape, modifications in ingot production are being made. It is hoped that within a few months, specimens will be tested in this high cycle range and examined both by fractographic and metallographic techniques.

Al-$CuAl_2$ System: At the time of the last progress report, more studies were proposed for the Al-$CuAl_2$ eutectic. These were planned to study the effect of growth rate upon mechanical properties and to investigate the kinking phenomenon of the lamellae observed in compression testing. A series of tests were conducted only to find upon metallographic examination that the specimens were off composition. Two more master heats of this alloy were prepared and difficulties encountered with macrosegregation. This problem has since been overcome and specimens of Al-$CuAl_2$ are now being unidirectionally solidified. Mechanical property evaluation of this material is expected in the near future.

ELEVATED TEMPERATURE PROGRAMS:

There are actually two projects under way in this category; one is an investigation of the stress-rupture properties of controlled Ni-Ni_3Nb eutectic and the other is a study of the thermal stability of the Al-Al_3Ni eutectic.

A. Ni-Ni_3Nb: In this project the Ni-Ni_3Nb eutectic is being studied as a potential high-temperature alloy composite. Previous work on this project has shown that the eutectic can be aligned to form a substantially parallel lamellar array of Ni and Ni_3Nb phase particles. The objectives of the work are to measure selected high-temperature mechanical properties and to develop an understanding of how and why the two phases, considered separately and in combination, influence the high-temperature mechanical properties.

Since the last progress report, in which only preliminary data were presented, substantial progress has been made. Suitable refractory boats (alundum) for production of unidirectionally specimens were received and some thirty-five five-inch controlled ingots were prepared at a solidification rate of 2.5 cm/hr. These are of such a size that 1/4" diameter tensile and stress-rupture specimens have been machined from them.

To date tensile properties have been determined at 25, 400, 600, 700, 800 and 1000°C. Ultimate tensile strength values observed are shown in Fig. 5A. At room temperature the stress-strain curves obtained with a clip-on extensometer consistently showed that the material deformed plastically before failure; total elongations between 1.75 and 2.00% were recorded. These observations confirm the preliminary results, mentioned in the last progress report, that the alloy does exhibit some ductility at room temperature. Due to experimental difficulties it was not possible to obtain elongation measurements in the hot tensile tests. However, qualitatively speaking, it is believed that the material retained or increased its ductility up to about 650°C, above which temperature a more brittle type of failure occurred. It will be noted that a change in slope of the tensile strength vs. temperature curve also occurs at about 650°C. These observations constitute one indication that a change in deformation and failure mode appears to be occurring in the temperature range of 600-700°C.

Concurrent with the experimental program on the aligned eutectic itself, another series of experiments has been initiated on the reinforcing phase, Ni_3Nb, to provide more fundamental data for a better understanding of the composite. A heat of the intermetallic compound has been prepared and used for a number of purposes. One set of experiments performed on this material consisted of a series of hot-hardness measurements. (The equipment for this--a Marshall unit--was received and put into operation during this reporting period.) The hot-hardness data were obtained to get an indication of the temperature dependence of strength of the intermetallic reinforcing phase. The results are shown in Fig. 5B in comparison with similar measurements on the aligned eutectic. It will be noted that the intermetallic maintained its room temperature hardness of Rockwell A72 up to about 700°C. At this point a sharp break in the curve was observed and the readings steadily declined to Rockwell A35 at 925°C. The eutectic alloy exhibited similar behavior; the hardness dropped only slightly between room temperature and 650-700°C and then decreased abruptly to Rockwell A23 at 925°C. These data then provide another indication that a change in mechanical behavior occurs in the vicinity of 600-700°C.

In the latter half of December the heat-resistant alloy grips necessary to perform the stress-rupture testing program were finally received from the manufacturer. No data are available yet but this phase of the testing program is now proceeding rapidly. Platinum gages are being used to record strain during testing.

A key part of the stress-rupture and tensile testing program is an evaluation, by means of optical metallography and electron fractography, of the deformation and failure modes which occur at different temperatures. This work is now in progress and is being supplemented by hot micro-bend test experiments being conducted in a jig constructed for the newly acquired hot-hardness tester. So far bend tests on the eutectic have been performed up to 500°C. Slip lines are observed in the intermetallic phase just as they were at room temperature (see previous progress report). It is hoped that this line of investigation can be pursued to at least 800°C to explore the behavior of the material in the vicinity of the transition on the tensile and hardness curves.

From all of these results obtained to date a paradox of sorts seems to be developing. Briefly stated it is as follows. At low temperatures, i.e., below 650-700°C, the intermetallic phase appears to be reinforcing the nickel even though positive indications of ductility in the reinforcing phase and the composite have been observed. Preliminary indications from the micro-bend tests in this temperature regime are that failure is initiated by cracks forming within the intermetallic due to multiple slip. Above about 700°C, where one might expect a material to exhibit more plasticity, the eutectic alloy appears to lose ductility, the hardness of the intermetallic rapidly decreases, yet a respectable hot tensile strength is observed in the composite. There could be many explanations for this behavior so the research is now focusing more sharply on the mechanisms behind this apparent transition in behavior around 600-700°C. It is hoped that the work will provide some fundamental concepts relating to elevated temperature behavior of composites in general.

Progress has also been made in another peripheral aspect of this program within the last six months. Since controlled eutectics in general exhibit not only a highly anisotropic microstructure but also, usually, a high degree of preferred orientation which will influence the properties, the crystallography of this system is being studied. Powder diffraction patterns of the intermetallic heat, Ni_3Nb, have been successfully indexed. (The crystallographic structure of the phase is not known with certainity. A literature search revealed that lines produced by the orthorhombic structure of Ni_3Nb had never been indexed before.) Having this information, it is now possible to proceed with a determination of the preferred orientation. The method devised several years ago by the principal investigator is being employed.

B. <u>Al-Al_3Ni</u>: This system, one of the first alloys in which the concept of making a fiber reinforced composite by the eutectic approach was demonstrated, is being used as a model system to investigate the effect of temperature on microstructural stability at temperatures near the eutectic temperature. Some work of this type has already been published on this alloy in the open literature and it is known that another paper is to appear soon. However all of this previous work suffers from the fact that the other investigators have not recorded all of the data necessary to truly establish the mechanism by which fiber coarsening occurs. Specifically, they have not measured the particle size distribution as a function of temperature and time. Since it is only from fiber diameter distribution measurements that one can hope to establish the rate controlling mechanism and thus a fundamental understanding of what is happening, these are the data being obtained on this project.

As mentioned in the previous progress report a series of specimens for heat treating studies was prepared at different growth rates (to give a series of specimens with different initial fiber sizes). But progress in obtaining the required quantitative metallographic data from the specimens has been slow because of a very frustating experimental difficulty which has been encountered. It is necessary, because of the small size of the reinforcing fibers of Al_3Ni, to make the quantitative size and size distribution measurements on high quality photomicrographs of replicas made in the electron microscope. Unfortunately an unexpected and inordinate amount of difficulty has been experienced in satisfactorily polishing and etching the microspecimens for replication. Many different etchants, electropolishing conditions and replication techniques have been tried in an attempt to consistently produce good photomicrographs. Quite recently these difficulties seem to have been overcome so it is hoped that progress on this phase of the research can take a huge step forward during the next reporting period. The sponsors can be assured that this project, although it has produced no significant results so far, is being pursued with the utmost vigor.

PHYSICAL PROPERTIES:

This project, initiated about nine months ago, is concerned with galvano-thermomagnetic effects in controlled Bi-Zn eutectic. The motivation behind the project is twofold; theoretically there are reasons to hope that galvano-thermomagnetic effects in highly anisotropic two phase materials consisting of a semi-metal and a metal can be capitalized upon to make very efficient energy conversion devices, and, secondly, very little work has been done to explore this possibility. The project is therefore largely exploratory in nature.

In the last six months progress has consisted of assembling and constructing the basic facility needed to measure various properties and in obtaining some of the first measurements. With respect to the apparatus itself it has been designed to measure thermal conductivity, electrical conductivity, the Seebeck coefficient or thermoelectric power, the Hall coefficient, the Nernst coefficient and the Ettingshausen coefficient. All can be measured in a magnetic field which is rotatable with respect to a specimen inserted in the apparatus and which is continuously variable--from zero up to about 25000 gauss. In addition the apparatus will permit the properties to be measured as a function of temperature from 77 to 400°K. Its construction is now essentially completed and consists of a specimen holder and thermocouple, power and sensing leads suspended within an evacuatable chamber which is inserted between the pole pieces of a magnet. The magnet and power supply, purchased from funds other than this grant, have been delivered and are presently being installed.

Preliminary measurements have shown that a miscalculation in the original design led to erroneous thermal conductivity measurements. Consequently an improved vacuum system which includes thermocouple-Pirani and ion gages capable of producing and measuring pressures down to 10^{-6} Torr has been constructed. This feature minimizes errors due to convection effects when measuring thermal properties. Also it has been necessary to redesign and build an improved specimen holder to minimize heat conduction through the various leads. The apparatus is now believed to be almost completely 'debugged'. A detailed description will be included in a future progress report or publication.

Actual experimental data obtained to date are still scattered and do not warrant reporting at this time. It is perhaps sufficient to say that quantitative metallographic measurements of the microstructure of the alloy have been made, preliminary determinations of the Seebeck coefficient have been made at 123 and 309°K, and that the thermal properties are presently receiving most of the attention because of the difficulty mentioned above. Calibration runs using pure bismuth as a standard are underway. Once they are completed it is planned to measure the various physical property coefficients of the aligned or controlled eutectic as a function of microstructural orientation, temperature and magnetic field. Hopefully the results will open the doors to new possibilities for energy conversion devices. Naturally attempts will be made to understand what is happening and why.

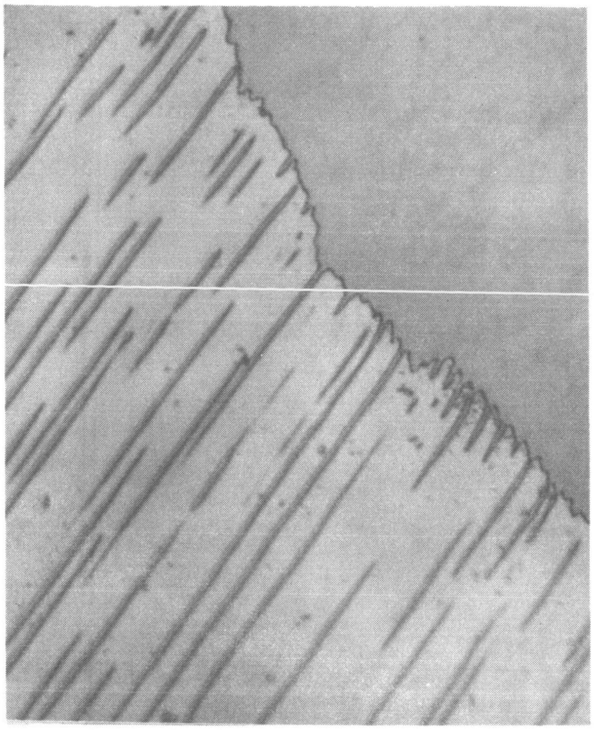


Fig. 1 - Metallographic section showing tensile type fracture with fiber failure. 1000X

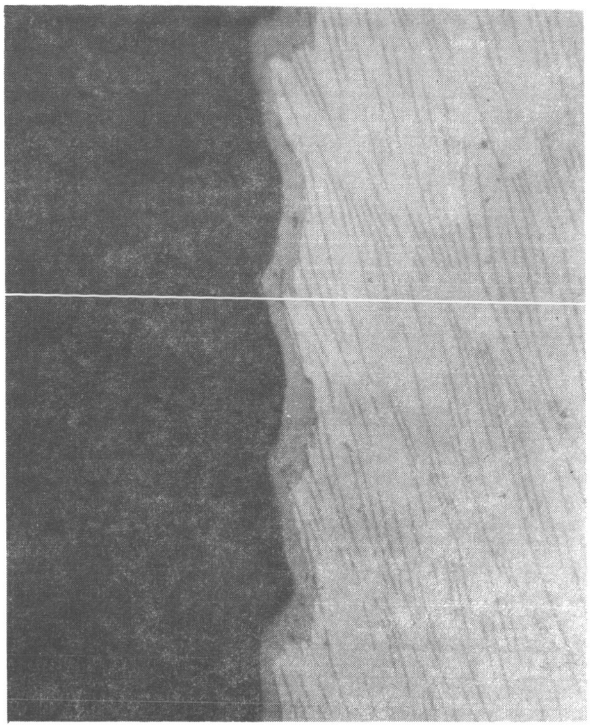


Fig. 2 - Typical fatigue fracture with alternating regions of interfacial failure and tensile failure. 500X

Fig. 3 - Fractograph revealing regions of elongated dimples and interfacial failure. 2,400X

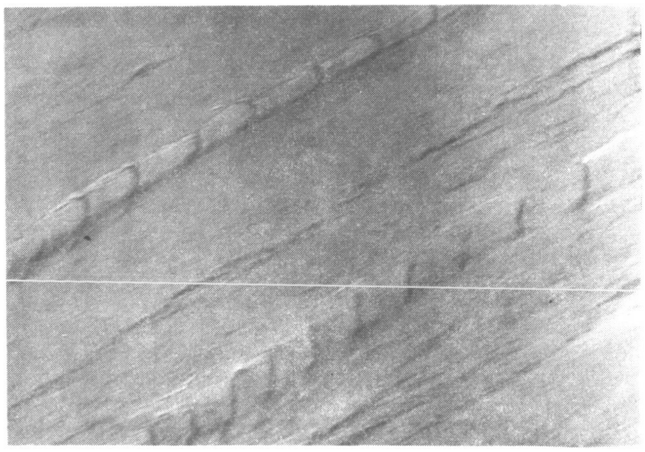
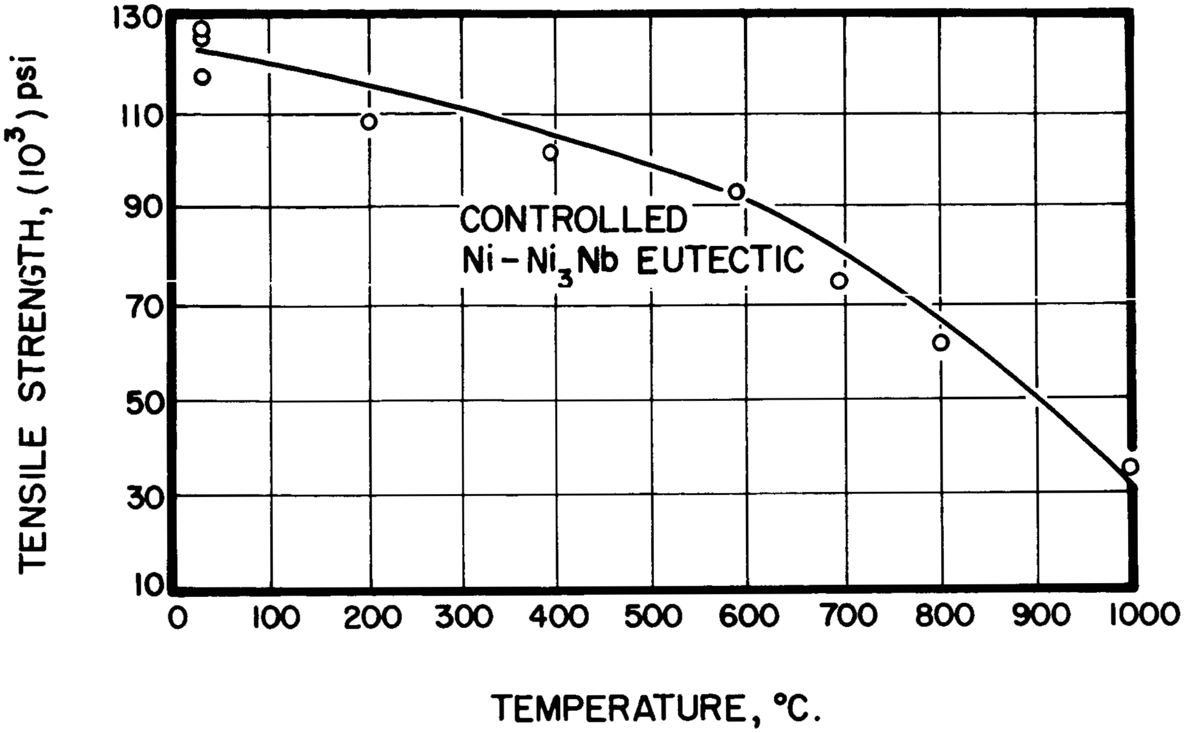


Fig. 4 - Evidence of fiber decohesion. 12,600X



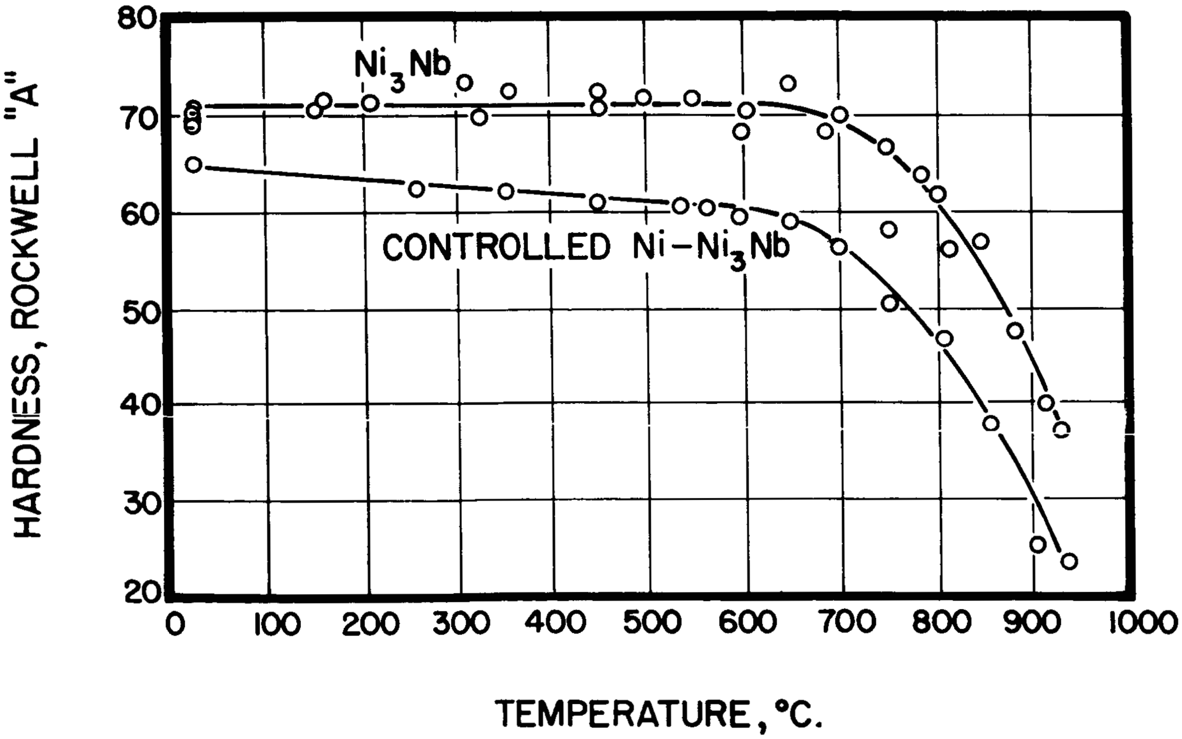


Fig. 5 - A, upper graph and B, lower graph.