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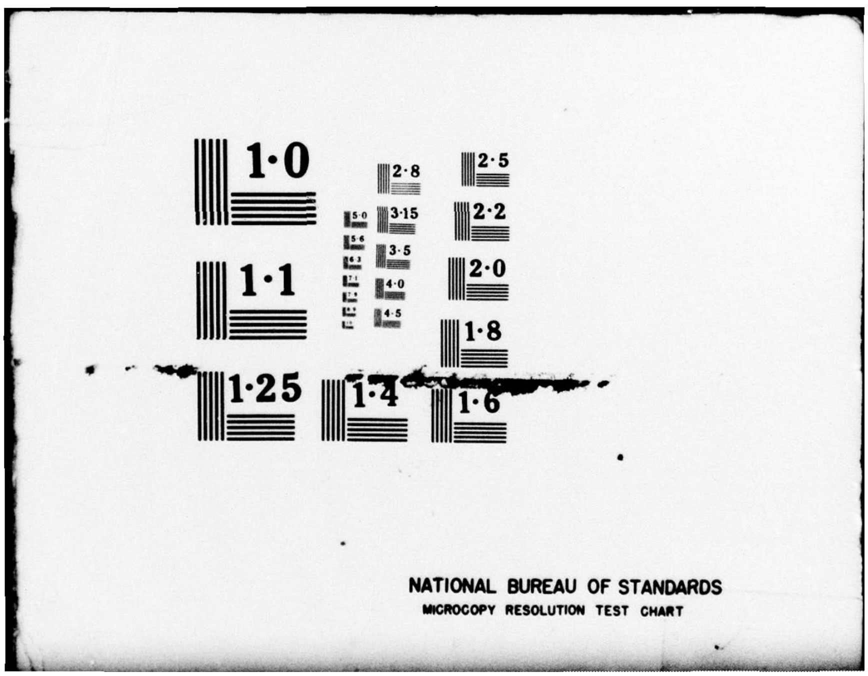
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Final Report

Office of Naval Research

Contract N00014-75-C-0503, NR 031-745

DEFORMATION AND FRACTURE OF CONTROLLED EUTECTICS CONTAINING INTERMETALLIC COMPOUNDS

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A. OVERALL OBJECTIVES OF RESEARCH

The principal aim of this research has been to define the microstructural features of aligned eutectics that can be expected to control tensile, creep and fatigue resistance. Efforts were centered upon eutectics containing intermetallic compounds because some of the principal alloys considered for high temperature structural applications ($\gamma/\gamma'-\delta$, γ'/γ-Mo, $\gamma'-\delta$, γ/γ'-TaC) contain one or more ordered intermetallic compounds, either as reinforcing composite phases or precipitate particles or both. Ordered alloys are characterized by excellent high temperature properties as a consequence of reduced diffusion rates within the compounds, which in turn reduces creep rate. In addition, the limited number of slip systems associated with long range ordered alloys leads to excellent fatigue resistance. Also, the identification of an alloy system (Ag_3Mg-AgMg), in which the degree of long range order in the Ag_3Mg phase could be altered by heat treatment, permitted a critical test of low temperature strengthening theories in eutectic composites. Finally, at the inception of this research program little attention was being paid by alloy designers to optimization of properties through control of microstructural features such as interphase spacing or distribution of a precipitated phase. Consequently, another major aim of this research has been to determine the role of interphase spacing and precipitate distribution upon mechanical properties of aligned eutectics.

B. ALLOYS CHOSEN FOR INVESTIGATION

During the early years of this program attention was centered on low-melting eutectic alloys: Ag_3Mg-AgMg, Ag-Ag_2Al and Al-Al_3Ni. Ag_3Mg-AgMg was the most important of these, because post-solidification heat treatments could be utilized to vary the state of long range order in the Ag_3Mg phase, thereby permitting direct assessment of the role of long range order in mechanical behavior. Another important reason for choosing these low-melting alloys was the ability to vary solidification rates, and therefore interphase spacings, λ, over wide ranges so that the dependence of tensile, creep and fatigue properties on λ could be assessed.

Since 1975 we have concentrated our efforts upon several high temperature, nickel-base eutectic composites,

Nitac - Ni,10Cr,5Al-14TaC

γ/γ'-δ (0%Cr) - Ni,21Nb,2.5Al

γ/γ'-δ (6%Cr) - Ni,20Nb,6Cr,2.5Al

A Ni,Cr,Al alloy corresponding to the matrix composition of Nitac also has been tested to provide composition data. Nitac alloys have fibrous microstructures with a low volume fraction of reinforcements (about 3%). The γ/γ'-δ alloys, on the other hand, are lamellar, with 30-37% reinforcing phase. Consequently, the two alloy types offered a sharp contrast in microstructure. However, both sets of alloys were amenable to post-solidification heat treatments to alter the γ' morphology present in the as-directionally solidified alloys. The latter was utilized as a fatigue test variable, together with interphase spacing for γ/γ'-δ, and test temperature, environment and frequency for Nitac. During the course of the investigation, research at Rensselaer on other fibrous aligned eutectics, notably Cotac (Co,Cr,Ni-TaC) and γ'/γ-Mo (Ni,Al,Mo), provided additional data on fatigue resistance as affected by microstructure. As a result, several review papers either on the general subject of mechanical behavior or on fatigue of eutectics were published and/or presented at technical meetings.

C. EQUIPMENT

During the course of this program several directional solidification furnaces were constructed. The first system, capable of temperatures to about 900°C by resistance heating was utilized for low-melting alloys. Later, two induction heated units were constructed.

Fatigue testing was carried out on three closed-loop Instron machines, with all tests in stress control on unnotched samples. All tests were conducted at 25°C in air (except for $Al-Al_3Ni$, which was tested to 410°C at several test frequencies in air), until early in 1977, when a vacuum chamber for high temperature testing was successfully completed. Since then considerable testing of Nitac was performed at 825°C in vacuum, at frequencies in the range 0.02 to 20 Hz. A limited number of constant-load creep tests also have been conducted in the range 800-950°C to identify deformation mechanisms and to determine the possible interaction between creep and fatigue processes at low cyclic frequencies.

SUMMARY OF RESEARCH ACCOMPLISHED

A. Effects of Interphase Spacing and Long Range Order on Mechanical Properties of Low-Melting Eutectics

Interphase spacing has been shown to markedly influence low temperature tensile properties, as well as fatigue resistance and high temperature creep behavior of $Ag_3Mg-AgMg$. Effects generally were more pronounced when Ag_3Mg was ordered, due to the influence of order in slip processes. Decreasing λ generally improves strength without impairing ductility; an exception was noted for the $Al-Ag_2Al$ alloy in which interphase boundaries are not strong barriers to plastic flow, so that decreasing λ actually reduced yield strength. This effect was ascribed to increasing grain size as solidification rate, R, increased. Previous to this work there had been nc report in the literature of eutectic grain boundaries affecting low temperature tensile properties. In one instance ($Al-Al_3Ni$) a very fine interphase spacing outweighed non-alignment of the microstructure in producing high fatigue resistance. A particularly striking effect of decreasing λ on increased fatigue strength was noted in elevated temperature tests on $Al-Al_3Ni$. Reduced interphase spacing also improved the room temperature fatigue behavior of $\gamma/\gamma'-\delta$, especially at high stress levels.

Creep resistance of $Ag_3Mg-AgMg$ was improved by refining λ, but much more significant was the suppression of creep when Ag_3Mg was ordered. The stress exponent, n, in the creep equation:

$$\dot{\varepsilon} = A\sigma^n e^{-Q/RT} \qquad (1)$$

was not affected by order, but decreased from about 14 at high stress levels to about 3 at very low stress levels. The activation energy, Q, for creep of the alloy increased from 43 to 79 kcal/mole with order in the Ag_3Mg phase.

B. Effects of Post-Solidification Heat Treatment on Fatigue Properties

Only limited success was achieved in improving room temperature fatigue properties of Nitac and $\gamma/\gamma'-\delta$ by heat treatment to control γ' size and distribution. However, at 825°C in vacuum a significant improvement in fatigue resistance of aged Nitac was noted, relative to the as-D.S. alloy. Heat treatments also influenced crack paths in fatigue of $\gamma/\gamma'-\delta$. The fatigue behavior of a Ni,Cr,Al "matrix" alloy was shown to be much inferior to that of Nitac at all stress levels.

C. Elevated Temperature Creep and Fatigue Properties of Nitac

Our vacuum system has been utilized for a series of fatigue tests at 825°C, frequency = 20 Hz, on Nitac in two microstructural conditions: as-directionally solidified and solution treated and aged. The aged material exhibited superior fatigue resistance, as had previously been observed at 25°C. Additional tests run in vacuum at 825°C at frequencies of 2, 0.2 and 0.02 Hz revealed a progressive decrease in fatigue resistance with decreasing frequency. Similar results had previously been obtained for $Al-Al_3Ni$; for both alloys a significant creep-fatigue interaction appears to be responsible, rather than an environmental effect. Creep tests were performed at 775°C-850°C in order to determine creep parameters in the same range of temperature and peak stress as used in the fatigue experiments. Data were anlyzed according to Equation 1. The activation energy, $Q = 152$ kcal/mole. The stress exponent, n, varied with temperature from 15 to 21. A high stress exponent is characteristic of eutectic alloys, as was previously described for Ag_3Mg-AgMg.

D. Fractographic Features

A notable feature of room temperature fatigue cracking in aligned eutectics is a strong tendency for slip-band cracking, also known as stage I-type cracking. This was particularly notable in Nitac (and Cotac) type alloys. Heat treatments had some effect on fracture morphology since fatigue striations were noted only in solution-treated Nitac, and then not in all tests. Striations were observed also in disordered Ag_3Mg-AgMg, but not in this alloy in the ordered condition, for which only slip-band cracking occurred. A strong tendency for deflection of fatigue cracks parallel to the tensile axis was noted both in Al-Al_3Ni and $\gamma/\gamma'-\delta$. Interphase boundary splitting, in fact, appeared to be a major source of the extremely high fatigue resistance displayed by $\gamma/\gamma'-\delta$ (0%Cr). The addition of 6%Cr produced a partially cellular microstructure under the same growth conditions, with markedly inferior fatigue properties. However, interphase boundary splitting was noted even in the 6%Cr alloy. Fatigue resistance of $\gamma/\gamma'-\delta$ (0%Cr) was shown to be superior to that of any other eutectic alloy for which tension-tension fatigue data have been reported in the literature.

Scanning electron fractography has revealed a distinct change in fatigue mode from stage I, crystallographic cracking at room temperature for all microstructural conditions to a dimpled fracture normal to the applied stress axis at 825°C; no striations were observed at elevated temperatures. Transmission electron microscopy has been utilized to determine dislocation substructures generated by both creep and fatigue experiments. A pronounced change in dislocation substructure appearance with test frequency in fatigue could be correlated with an increased creep component at low frequencies. These results, taken together with fatigue life vs. frequency data, are strong evidence that creep-fatigue interactions play a dominant role in elevated temperature fatigue experiments.

Fractographic features for tensile and creep tests also were recorded during this program with the results published in a review paper.

TECHNICAL REPORTS

Contract N00014-67-A-0117-0010, NR 031-745

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

The results of research performed under this contract on the following aligned eutectics are summarized: Ag_3Mg-AgMg, Al-Ag_2Al, Al-Al_3Ni, Ni,Cr,Al-TaC (Nitac) and Ni,Nb,Cr,Al ~~(γ/γ'-δ)~~. Tensile, fatigue and creep experiments have shown the importance of refining interphase spacing in producing increased strength. γ/γ'-δ with no Cr is shown to have the best tension-tension fatigue resistance of all aligned eutectics.

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