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HIGH TEMPERATURE STABILITY, INTERFACE BONDING, AND MECHANICAL BEHAVIOR IN β-NiAl AND Ni_3Al MATRIX COMPOSITES WITH REINFORCEMENTS MODIFIED BY ION BEAM ENHANCED DEPOSITION

Progress Report

for the period June 1, 1991 - May 31, 1992

D. S. GRUMMON

Michigan State University
Department of Metallurgy, Mechanics and Materials Science
East Lansing Michigan 48824-1226

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INTRODUCTION

Research funded by the U. S. Department of Energy office of Basic Energy Sciences, under grant # DE-FG02-85ER45205 entitled "Softening Mechanisms and Microstructural Instabilities During High Temperature, Low Cycle Fatigue of Ni, Ni_3Al and their Metal Matrix Composites", was begun under professor Gunter Gottstein in 1985. The initial 3-year grant was renewed in 1988. In connection with the departure of Gottstein from MSU in 1989, professor D. S. Grummon was added as a co-principal investigator and assumed day-to-day responsibility for the project. In 1991, following peer review, the grant was renewed under the new title "High Temperature Stability, Interface Bonding, and Mechanical Behavior in NiAl and Ni_3Al Matrix Composites with Reinforcements Modified by Ion Beam Enhanced Deposition", for the three-year period beginning June 1, 1991, under the direction of professor Grummon acting as a single investigator. The following progress report summarizes activity and progress made during the first year of the new project.

Progress Summary: June 1, 1991 - May 31, 1992

MODIFICATION OF REINFORCEMENTS FOR ALUMINIDE MATRIX COMPOSITES

Development of Experimental Apparatus. Modification of our high-vacuum film deposition system to allow for ion beam assisted ion sputter deposition and ion assisted vapor deposition have been completed. In addition to the 3-cm Kaufman gun installed for use as an ion sputtering exciter, a second ion gun (an Anatech 5-cm source designed for acceleration of reactive species) has been installed. A custom-designed 3-kW rod-fed electron beam heated evaporation source has also been acquired for use with the 5-cm source in ion-assisted physical vapor deposition experiments. The relatively high inert gas throughputs required for operation of the ion sources, and the stringent vacuum requirements of the electron beam source, have necessitated a significant increase in pumping speed. This has been accomplished through the addition of an APD8-S cryopump, specially designed for high argon gas throughput. Additional equipment of utility to the project has been acquired, namely a particle surface area measurement system, and a high temperature dilatometer, both donated to the PI's lab by Ford Motor Company. A simple thermal cycling device has been constructed which will be refined and partially automated for future experiments.

Initial trials and calibration of a Dow Interfacial Testing Machine for single-fiber pushout measurements of interfacial shear strength (ISS) have been completed, and early results have been presented. It has been concluded that this machine is adequate for testing of ISS levels in composites with the relatively thin (20 μm) FP-alumina fibers, but may not be able to achieve sufficient pushout force to debond larger SAPHIL fibers. Further development of this technique, in addition to experimentation with 3-point bend test methods for ISS estimation at elevated temperature, will be required.

Procedures for in-house hot isostatic pressing of composite specimens have been devised which will use thin-wall stainless-steel tubes sealed after evacuation by a crimping method. This simplified approach will be adequate for production of small button-head specimens suitable for use in our high-temperature MTS load frame.

With respect to the latter, we have conducted some initial experiments in the production of alumina diffusion barriers by reactively sputtering Al_2O_3 onto planar substrates of the TZ-molybdenum alloy used in high-temperature components of the MTS load frame. These films successfully inhibited diffusion bonding of grip components and will allow practical tests to be performed, with hot grips, at temperatures above 1400 °C.

Acquisition of Materials. Several kilograms of β-nickel aluminide have been obtained in both monolithic form (a 3-inch hot extruded bar) and as powders in both 80 and 325 mesh particle sizes. A spool of DuPont FP-alumina fibers is on hand, as well as a small quantity of SAPHIL single-crystal sapphire fiber. When additional SAPHIL fibers have been acquired, sufficient materials will be available for the next few year's experimentation. Aluminum and nickel sputtering targets have been fabricated and tested.

Diffusion Bonding of Al_2O_3-βNiAl Composites. In preparation for a series of experiments with surface modified Al_2O_3 reinforcements in βNiAl, we have conducted a series of diffusion bonding experiments (using as-supplied FP fibers) to establish optimum processing conditions for these materials with regard to time, temperature and bonding stress. Table I, below, summarizes results of the tests, in which an optimum protocal was established which calls for diffusion bonding with a stress of 15 MPa at 1673K for 4 hours in vacuum.

TABLE I

TRIAL DIFFUSION BONDING CONDITIONS FOR βNIAL-AL_2O_3 COMPOSITES

Run	Temperature, K	Stress, MPa	Time, hrs.	Results
1	1573	35	2	Overstressed: breakup, creep
2	1573	15	2	Incomplete diffusion bonding
3	1623	15	4	Incomplete diffusion bonding
4	1673	15	4	Good bonding
5	1673	9	4	Incomplete diffusion bonding

Surface Modification Experiments with Al_2O_3 Fibers in β-NiAl. FP alumina fibers have been prepared with ion sputtered surface films having a 100nm thick alumina layer deposited by reactive ion beam assisted deposition, to which were added discrete layers of ion sputtered pure aluminum, and and finally, pure nickel. A schematic view of a typical modified fiber cross-section is shown in Figure 1. These fibers were composited with thin slabs of extruded β-NiAl and hot-pressed under conditions similar to Run #4 in Table 1.

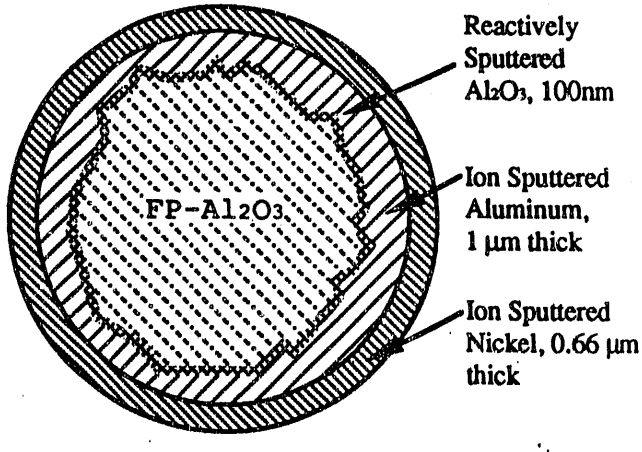

Figure 1.

The specified Ni:Al thickness ratio of 0.66:1 is such as to produce the β-aluminide stoichiometry upon reaction of the elemental constituents.

After hot-pressing, the composite specimens were subjected to approximately 70 thermal cycles between 315K and 1000K, using quartz-lamp radiant heating (in air), and forced-air cooling. Thin sections of the of both as-pressed and thermally cycled composites were prepared with a low-speed diamond saw and further ground to thicknesses of approximately 100 mm. Interfacial shear strength of the fiber-matrix interface was determined for modified and control fibers using an Interfacial Testing System (ITS, developed by Dow Chemical Co.). Tensile strength of control and modified fibers was measured, and surface morphological features were studied by scanning electron microscopy of fibers exposed by electrolytic removal of the surrounding matrix material. Auger electron spectroscopic scans were carried out to determine the composition of the composite system in the vicinity of the fiber-matrix interfaces.

Both control (uncoated) and modified fibers showed very high ISS levels in the as-pressed condition, before thermal cycling, which were too high to be quantified with the Dow ITS apparatus. However, results of tests on thermally cycled specimens indicated that room temperature interfacial shear strength (ISS) in FP-alumina fiber - NiAl composites was enhanced significantly when the layered surface film was present during composite processing. Control composites showed ISS values of approximately 46 MPa, whereas specimens with modified fibers showed ISS levels exceeding 250 MPa. These data are considered preliminary since they represent our first attempt to use the Dow ITS apparatus to make these measurements, but subsequent SEM study of fiber surface morphology suggests a possible mechanism for the observed increase: Fibers which possessed the bi-phase metallic coating prior to diffusion bonding were found to have significantly rougher surfaces than those which were composited in the as-supplied condition. Furthermore, it was clear that the alumina was subject to substantial microstructural instability during high-temperature processing, resulting in creep and grain growth of both control and modified fibers.

We believe that the observed difference in surface roughness can be attributed to differences in the local stress-state experienced by the fibers in the control and modified conditions. The uncoated (control) fibers experienced, during diffusion bonding, a stress-state having a large deviatoric component, accentuated by the requirement that matrix material flow around the diameter of the fiber as the bonding process proceeded. The coated fibers, on the other hand, were effectively contained in a sputtered metal 'vessel', in which there may have occurred a transient liquid phase, and which would , in any case have, conformed intimately to the irregular surface of the fiber. The latter effect may have mitigated unfavorable wetting conditions between Al_2O_3 and NiAl, but more importantly, the soft metal encapsulation may have rendered the local stress-state at the fiber surface more hydrostatic than was the case for uncoated fibers, reducing the tendency to smooth out surface asperities by plastic flow. The enhanced surface roughness of the treated fibers (or, more accurately, the degradation of roughness in the uncoated fibers) may account for the observed differences in ISS level. It is not yet clear to what degree fiber tensile properties were affected by the coating and further experiments are in progress to confirm the observed ISS enhancement, to quantify fiber surface roughnesses, and to determine more accurately the effect of the coating on strength of the fibers. However, these

initial observations were unexpected and interesting, and may lead to approaches to fiber surface modification which were not originally anticipated.

Creep in Ni_3Al

Constant true stress compressive creep tests have been completed at 80 and 160 MPa at 1000 °C; 20, 40, 80 and 160 MPa at 1100 °C; 6, 10, 20, 40 and 80 MPa at 1200 °C; and 10, 20 and 40 MPa at 1300 °C. It was found that, at 1000 oC, the primary creep stage exhibited inverse transient behavior which shifted to regular transient creep at decreasing stress and increasing temperature. Few of the tests attained a sustained steady-state creep condition, but rather displayed a creep-rate minimum followed by a second transient stage, associated with the onset of dynamic recrystallization. The latter was confirmed in TEM examination of specimens from interrupted tests, where subgrain formation and grain-boundary bulging were observed.

If the minima in the creep rate curves were taken as quasi steady-state deformation rates, the material could be shown to exhibit power-law behavior with a stress exponent of 3.4 and an activation energy of 3.4 eV per atom. The structures evolved a random distribution of dislocations, indicative of Class-I alloy behavior. However, at stress levels below 20 MPa, a stress exponent of 4.2 was obtained and subgrain formation characteristic of Class-II alloys was observed, indicating a possible transition from Class-I to Class-II behavior at low stress. A summary of the kinetic data is shown below in Figures 2 and 3.

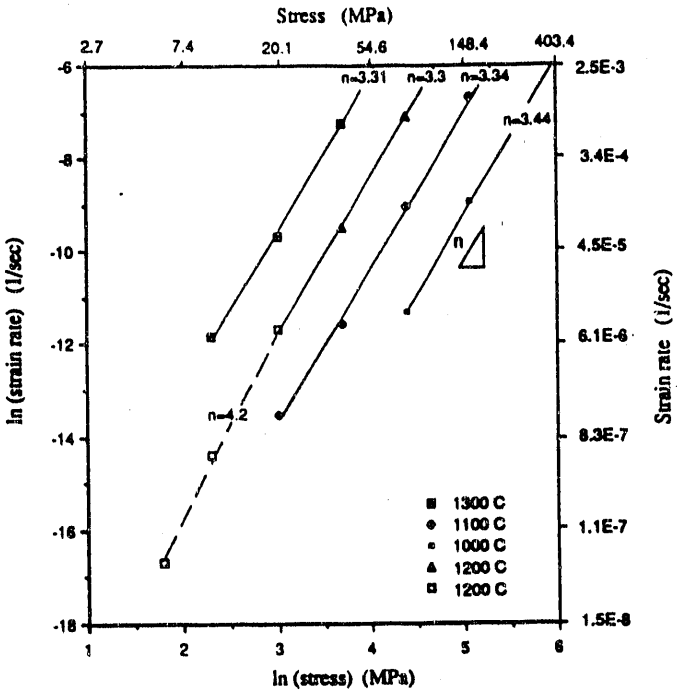

Figure 2. Stress Dependence of the minimum creep rate in polycrystalline Ni_3Al

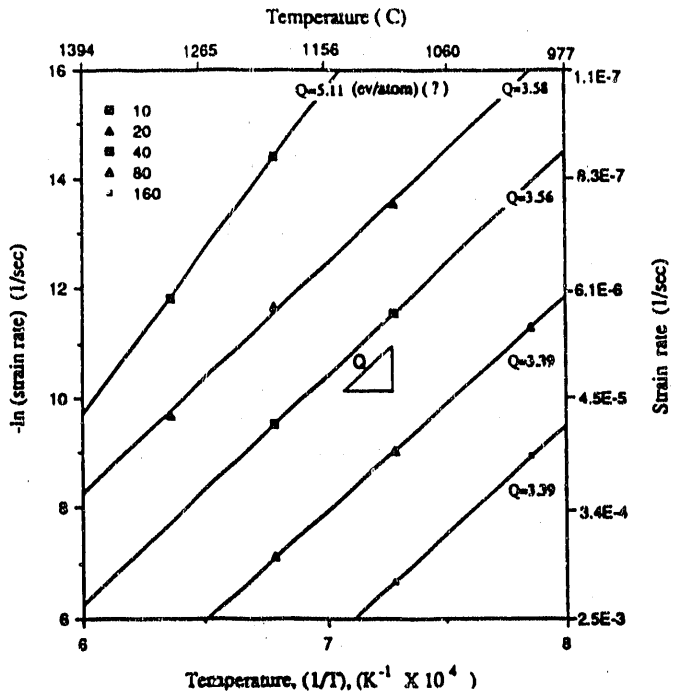

Figure 3. Temperature dependence of the minimum creep rate in polycrystalline Ni_3Al

Microstructures of the crept specimens varied from partially recrystallized structures with bi-modal grain size distributions, through fine-grained recrystallized structures, to coarse-grained structures with wavy grain boundaries. The grain size of the recrystallized structures were found to be a function of the applied stress, but was

independent of the test temperature. The stress-dependence of grain size followed a Hall-Petch type power law with a stress exponent of -0.68.

Publications Acknowledging DOE Support

Papers:

1. D. S. Grummon, Z. Cai and R. Schalek, "Transient Surface Reactivity of Ion Beam Modified Al_2O_3 Particles in β-NiAl Matrix Composites", to be published in *Structure and Properties of Metal-Ceramic Interfaces*, Proc. Symp. Cincinnatti OH., Oct. 1991, D. S. Grummon, K. N. Subramanian and J. Singh. Eds., TMS 1992 [in press].

2. C. Lee, D. S. Grummon and G. Gottstein, "Microstructure and Interface Behavior in Diffusion Bonded Ni_3Al+B Matrices Containing Continuous AL_2O_3 Fibers", in *Intermetallic Matrix Composites*, D. Anton, P. Martin, R. McMeeking and D. Miracle, Eds., Mat. Res. Soc. Symp. Proc. 194, pp. 301-306 (1990).

3. C. Lee and D. S. Grummon, "Diffusion Bonding of Continuous SiC and Al_2O_3 Fibers in Boron Doped Nickel Aluminide", in *Structural Composites: Design and Processing Technology*, American Society for Metals, pp. 585-592 (1990).

Presentations:

D. S. Grummon, Z. Cai and R. Schalek, "Transient Surface Reactivity of Ion Beam Modified Al_2O_3 Particles in β-NiAl Matrix Composites", TMS Fall meeting, Cincinnati OH., Oct. 1991.

G. Gottstein, D. S. Grummon, C. S. Lee and D. Ponge, "Large-Strain Deformation Behavior of Ni_3Al+B at High Temperatures", ICSMA-9, Haifa, Israel, June 1991.

Master's Theses:

Z. Cai, "Thin Film Al_2O_3 Diffusion Barriers by Magnetron Sputter Deposition", MS, May, 1992.

Doctoral Theses:

C-S. Lee, "High-Temperature Behavior of Ni_3Al Under Compressive Stress: Creep and Diffusion Bonding", Ph.D., September, 1991.

END

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